


# Chen Northern, Inc.

Analytical chemistry laboratory services for the IWD RI/FS will be provided by Wadsworth Alert Laboratory. Chen-Northern will perform the laboratory work associated with the geochemical analyses discussed in the hydrogeological investigation. Daniel B. Stephens and Associates will perform the physical property analysis work.

## **QUALITY CONTROL MANUAL CHEMICAL/INDUSTRIAL HYGIENE DIVISION OCTOBER 1989**

**Chemistry Laboratory**

Prepared by:  Laboratory Manager

Date: June, 1991

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## **1.0 PURPOSE**

The purpose of this manual is to provide written policies and procedures for testing services provided by the Chemical and Industrial Hygiene Division of Chen-Northern, Inc. These written policies and procedures communicate to all employees the standards of performance expected within our organization. These policies and procedures are consistent with those established by Chen-Northern, Inc. in other areas of practice. Our quality control system covers personnel, equipment, and supervision. Established written guidelines are provided for the development and training of our personnel. Equipment maintenance and calibration schedules are covered.

A quality control coordinator has been designated to monitor the program and report discrepancies to both the Chemical Laboratory Manager and the Area Vice-President.

Our quality control manual serves as a basis for conformance audits by our clients, federal agencies, and certification organizations.

## 2.0 OBJECTIVES

It is our objective to document the quality of the data generated by our testing services, and thus to maintain a reputation of quality service, including timely, and within budget expectations. We intend to meet these objectives by charging a reasonable fee to our clients and by making a profit for the owners of Chen-Northern, Inc.

Specific objectives of our standards of performance are as follows:

- ♦ To develop, review, and update laboratory practices and routine methodologies. This will encompass, among other things, analytical procedures, sampling and sample preparation, and personnel training.
- ♦ To monitor and take any corrective action required to maintain a performance level consistent with our guidelines, client needs, and/or regulatory agency requirements.
- ♦ To utilize personnel who are trained for the tasks assigned and to provide the supervision and expertise necessary to determine acceptable variations from standard procedures and practices of recognized chemical laboratory techniques.
- ♦ To inventory, maintain, and calibrate testing equipment used in our business.
- ♦ To review all laboratory data before results are given to the client.
- ♦ To participate in inter-laboratory and other round robin evaluation programs to monitor the consistency and level of quality within the chemical laboratory.

### 3.0 ORGANIZATION

Northern Engineering and Testing, Inc. was founded in 1958 in Great Falls, Montana, serving primarily the Northwest. Major offices were subsequently established in Boise, Idaho; Billings, Montana; Great Falls, Montana; and Salt Lake City, Utah; branch offices in Casper, Wyoming; Pocatello, Idaho; Tri-Cities, Washington; and Evanston, Wyoming serve outlying regions in those areas. Chen and Associates was founded in 1961 in Denver, Colorado serving the Rocky Mountain region. Major offices were subsequently established in Casper, Wyoming; Salt Lake City, Utah; Phoenix, Arizona; and San Antonio, Texas; and branch offices in Colorado Springs and Glenwood Springs, Colorado; Rock Springs and Cheyenne, Wyoming; and Elko, Nevada. In 1987 both firms were acquired by Huntingdon International Holdings (HIH). Chen-Northern, Inc. was founded in 1988 as a result of a merger of Northern Engineering and Testing, Inc. and Chen & Associates, headquartered in Denver, Colorado (see attached organizational chart, Figure 3-1).

The services provided by the chemical laboratory include solid waste analysis, trace metal analysis, air quality monitoring, water and wastewater analysis, mining-related soils analysis and asbestos identification.

As our clients require testing and/or the analysis of materials outside our areas of expertise, we will then employ affiliate laboratories or consultants who have demonstrated expertise and qualifications consistent with the quality-of-service precepts of Chen-Northern.

The organizational structure of the Chemical and Industrial Hygiene Division requires the placement of the quality control function in the laboratory and this is shown in diagram form in Figure 3-2.

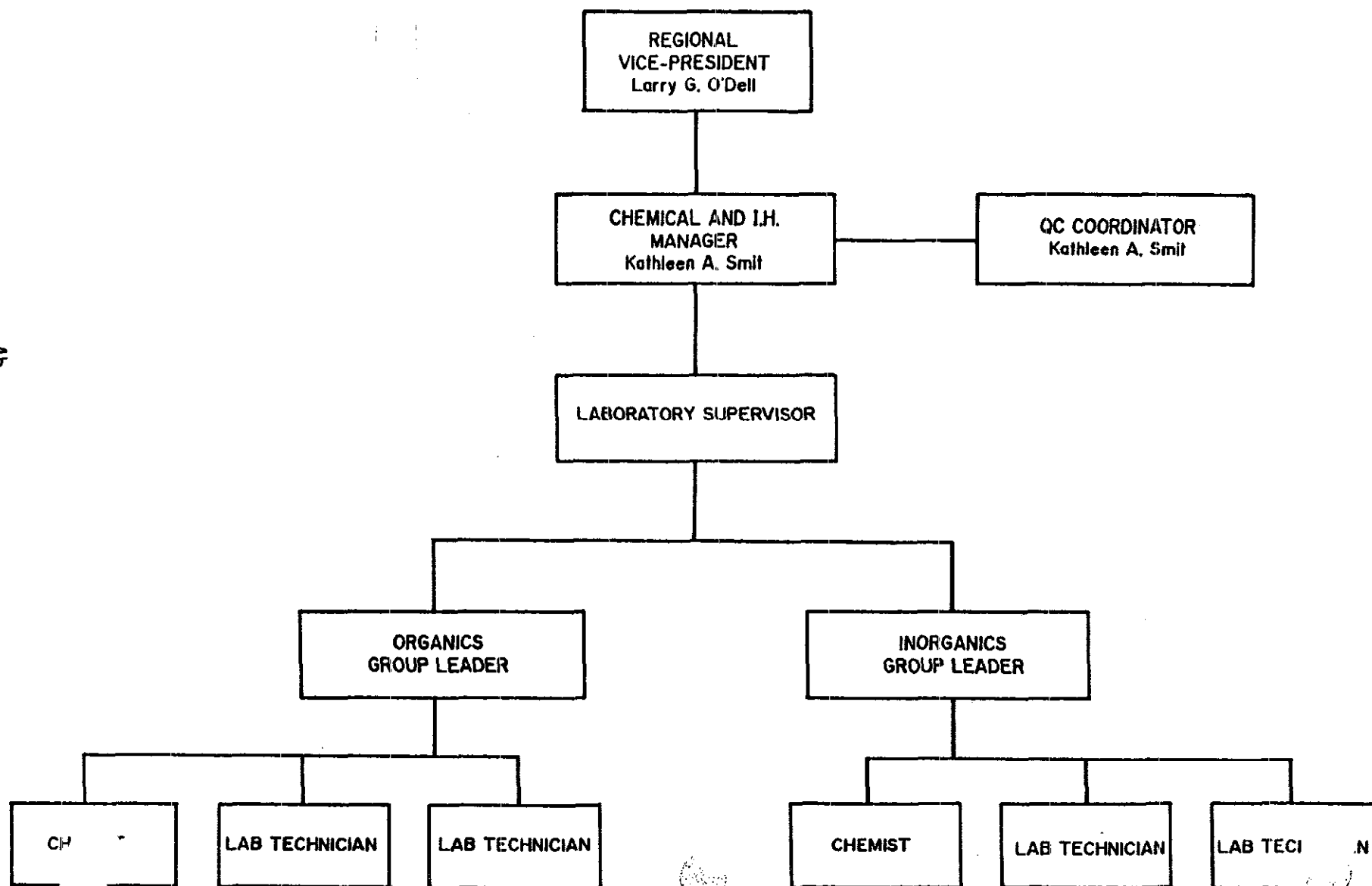
The Quality Control Coordinator is responsible for monitoring, recordkeeping, statistical controls and any other function required to maintain the quality assurance system. The Quality Control Coordinator is also responsible for recommending measures to ensure the fulfillment of those objectives of management relating to quality control and to carry out these objectives in an efficient and economical manner.



RESERVED  
FOR  
FUTURE  
USE

FIGURE 3-2

QUALITY CONTROL LINE AND STAFF  
ORGANIZATIONAL CHART



#### **4.0 FACILITIES**

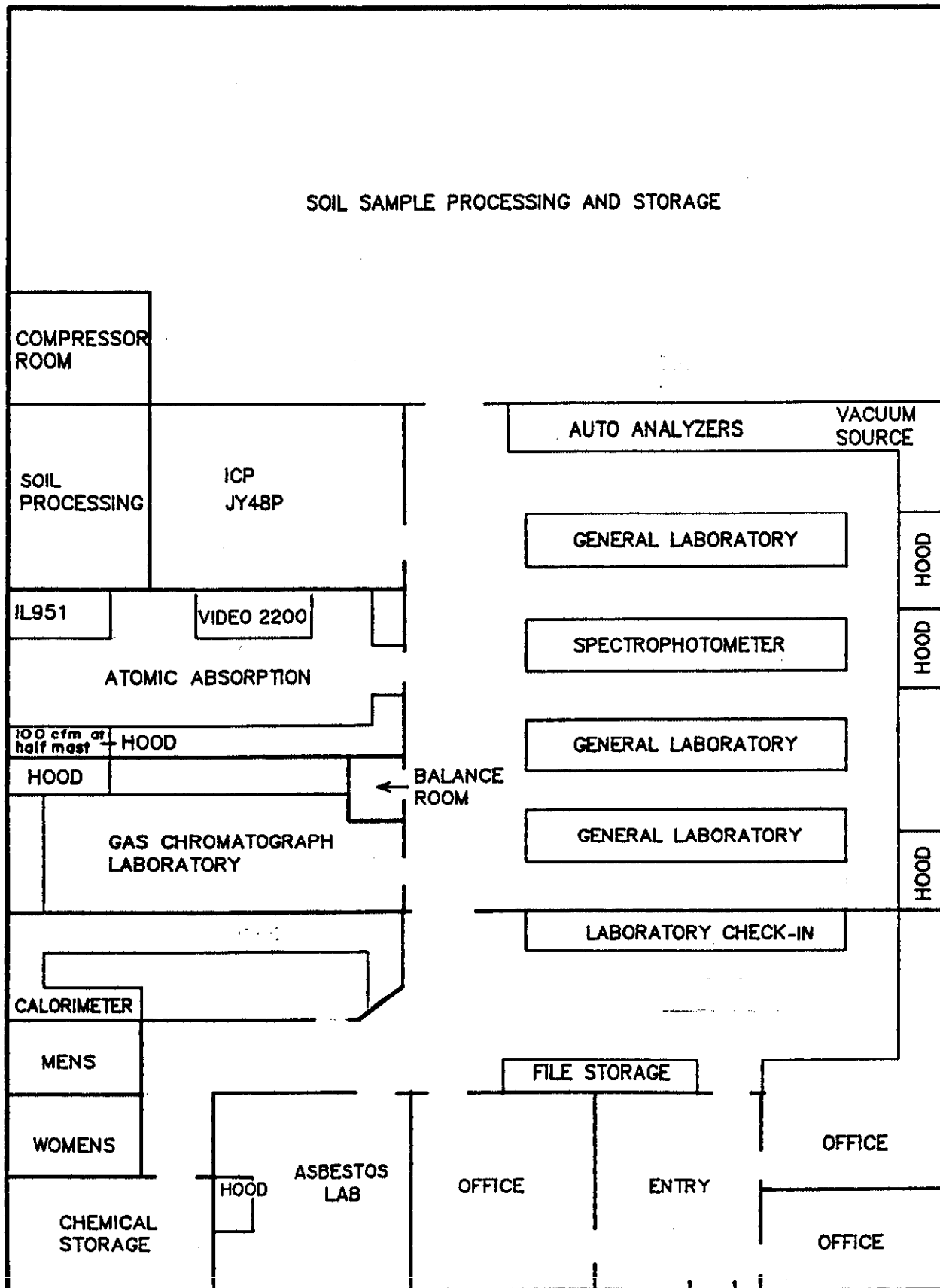
The Billings laboratory has a total of 3500 square feet of floor space assigned to laboratory service. The laboratory staff uses the services of the clerical staff that is located in the engineering office. An area of 1500 square feet is used as additional storage area. Figure 4-1 shows the layout of Chen-Northern's facility, including labeled areas for specific laboratory analysis.

The air quality of the laboratory is controlled by the use of ventilation. The laboratory is equipped with five hoods having an air flow of approximately 750 cubic feet per minute to provide ventilation. These hoods are used for exhausting toxic and hazardous fumes and odors. All digestion procedures are completed under the hoods. Their locations are shown in Figure 4-1. There are also canopy hoods for general elimination of hazardous and noxious vapors from the Atomic Absorption and Inductively Coupled Plasma Units.

The laboratory facility is equipped with two forced air heating systems. Air conditioning is also provided by use of two central units.

The noise level in the laboratory is minimized and controlled in order to provide a working environment compatible to the activities associated with wet instrumental and physiochemical methods of analysis. The level of tolerable noise does vary, depending upon the type of activity taking place.

FIGURE 4-1



## **5.0 RECORDKEEPING, CHAIN OF CUSTODY, DOCUMENT CONTROL**

Records are maintained in the chemical laboratory which identify the sample, request the prescribed analysis, establish the chain of custody, and review and document the report. Records are also maintained on personnel qualifications, equipment status, and quality control reports.

Upon receipt of a sample in the laboratory, it is forwarded to the laboratory manager's representative. The identification information accompanying the sample is recorded on the Sample Log Index (Figure 5-1) and is assigned a number. This is accomplished using the laboratory information management system (LIMS).

A work order (Figure 5-2) and the appropriate tracking charts are completed with the sample identification, laboratory number, and a description of the work requested.

The white copy of the work order is posted in the laboratory with any packing slips, purchase orders, chains of custody or letters of transmittal that accompanied the sample upon delivery. The yellow copy is kept in a time file for scheduling purposes. The pink copy is kept by the business office for invoice verification.

Upon completion of the analyses, the worksheets are checked by a technician and results are entered into the LIMS. The data is summarized in report form (Figure 5-3) by the LIMS and reviewed by the laboratory manager.

The report, accompanying paper work, and work order are compiled by the laboratory manager for publishing and billing. The laboratory manager reviews the published report and accompanying invoice for billing and signs the report for distribution.

The number of requested copies are mailed to the client and the original and the worksheets are placed in the project file. The original report is retained indefinitely and the data worksheets are kept on file three years following the date of the report.

The sample is maintained in the laboratory for seven days after the report has been sent to the client. The sample is either disposed or retained if requested in writing by the client.

FIGURE 5-1

# SAMPLE LOG SHEET

[illegible]

Date Received \_\_\_\_\_ Collected By \_\_\_\_\_

[illegible]

DESCRIPTION				QUANTITY	UNIT PRICE	TOTAL

Date Completed: \_\_\_\_\_



FIGURE 5-3

Client:  
Identification:  
Laboratory Number:  
Date Sampled:

Date  
Job No.  
Sheet

Date Analyze:

pH, standard units:  
Conductivity, umhos/cm:  
Total Dissolved Solids  
(at 180 C), mg/l:  
Sodium Adsorption Ratio (SAR):

CATIONS

Total Hardness as CaCO <sub>3</sub> :	mg/l	meq/l
Calcium as Ca:	mg/l	meq/l
Magnesium as Mg:	mg/l	meq/l
Sodium as Na:	mg/l	meq/l
Potassium as K:	mg/l	meq/l
Total Cations:		meq/l

ANIONS

Total Alkalinity as CaCO <sub>3</sub> :	mg/l	meq/l
Bicarbonate Alkalinity as HCO <sub>3</sub>	mg/l	meq/l
Carbonate Alkalinity as CO <sub>3</sub> :	mg/l	meq/l
Hydroxide Alkalinity as OH:	mg/l	meq/l
Chloride as Cl:	mg/l	meq/l
Fluoride as F:	mg/l	meq/l
Nitrate + Nitrite as N:	mg/l	meq/l
Sulfate as SO <sub>4</sub> :	mg/l	meq/l
Total Anions:		meq/l
Cation-Anion Difference:		meq/l

Ortho-Phosphate as P: mg/l

TRACE ELEMENTS, DISSOLVED

Aluminum as Al:	mg/l
Boron as B:	mg/l
Cadmium as Cd:	mg/l
Copper as Cu:	mg/l
Iron as Fe:	mg/l
Lead as Pb:	mg/l
Manganese as Mn:	mg/l
Mercury as Hg:	mg/l
Selenium as Se:	mg/l
Silver as Ag:	mg/l
Vanadium as V:	mg/l
Zinc as Zn:	mg/l

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## 6.0 QUALITY CONTROL POLICIES

Policies which are implemented by the chemical laboratory to achieve and support the quality objectives are listed in this section. Details for implementing these policies appear in later sections of this manual.

Administrative and technical review of laboratory reports and records to assure validity and uniformity is provided by the assigned QA coordinator, the technician generating the data and the laboratory manager.

Complete and current analytical methods and procedures and analytical instrument operating instructions are made available to laboratory personnel. Data and calibration records are updated upon their generation by laboratory personnel.

Only skilled and trained personnel are employed. The equipment used in the laboratory is maintained and calibrated on a regular basis as described by the method used for quantification of the analyte of interest. All chemicals, reagents, and precision equipment used in the laboratory are certified or standardized and documented on a regular basis.

The following flow diagram (Figure 6-1) indicates the quality control routine used by Chen-Northern. This flow diagram governs the actions of the technicians as they analyze samples for any parameter. A generalized rule for all tests includes the following salient points:

1. Standards traceable to National Bureau of Standards or primary standards as defined by the American Chemical Society and at least one blank (reagent blank) shall be run with any number of samples to be analyzed. In the event

more than 20 samples are analyzed in one batch, the standards shall be verified after each set of 20 analyses. Internal standards are not currently used in our operation.

2. Each inorganic batch shall include 10 percent duplicate analyses, 10 percent spike analyses and at least one standard reference material. Reference material concentration recoveries shall be compared to accuracy acceptance criteria. Reference material concentrations shall be plotted on control charts for identification of bias.
3. Each organic batch shall include at least one daily spike and 10% duplicate analysis. Appropriate blank analyses will be accomplished as needed. For volatile organics, the recovery of a surrogate material will be evaluated for each purged sample.
4. All raw data such as tare weights, titration volumes, absorbances, peak heights, fiber counts, etc. will be recorded in the laboratory notebook. Correlation coefficients, slopes and y intercepts shall be recorded for Beer's law curves used for data generation.
5. Dates and technician initials shall be recorded with raw data. Notes of unusual circumstances or special sample treatments shall be entered in the lab notebook.
6. Raw data shall be converted to concentration units by using the calculations given in the appropriate written method. Deviations from this shall be recorded.

In an effort to verify control and demonstrate capability as an analytical laboratory, Chen-Northern subscribes to the following quality audit programs:

- ♦ EPA - SDWA Drinking Water Supply Audit - Biannually
- ♦ EPA - NPDES Wastewater Source Audit - Biannually
- ♦ NIOSH - PAT Program - Asbestos - Quarterly
- ♦ NBS - Asbestos Identification Audit - Biannually
- ♦ Utah State University - Soil Analysis Round Robin - Annually

Quality control acceptance criteria for precision and accuracy is generated using data produced in our laboratory. For precision, the acceptable range of duplicate concentrations is determined by calculation of critical differences of data produced in the past. Using the following equation, a critical difference or "Range of duplicate" is established for each range of concentration of any particular analyte.

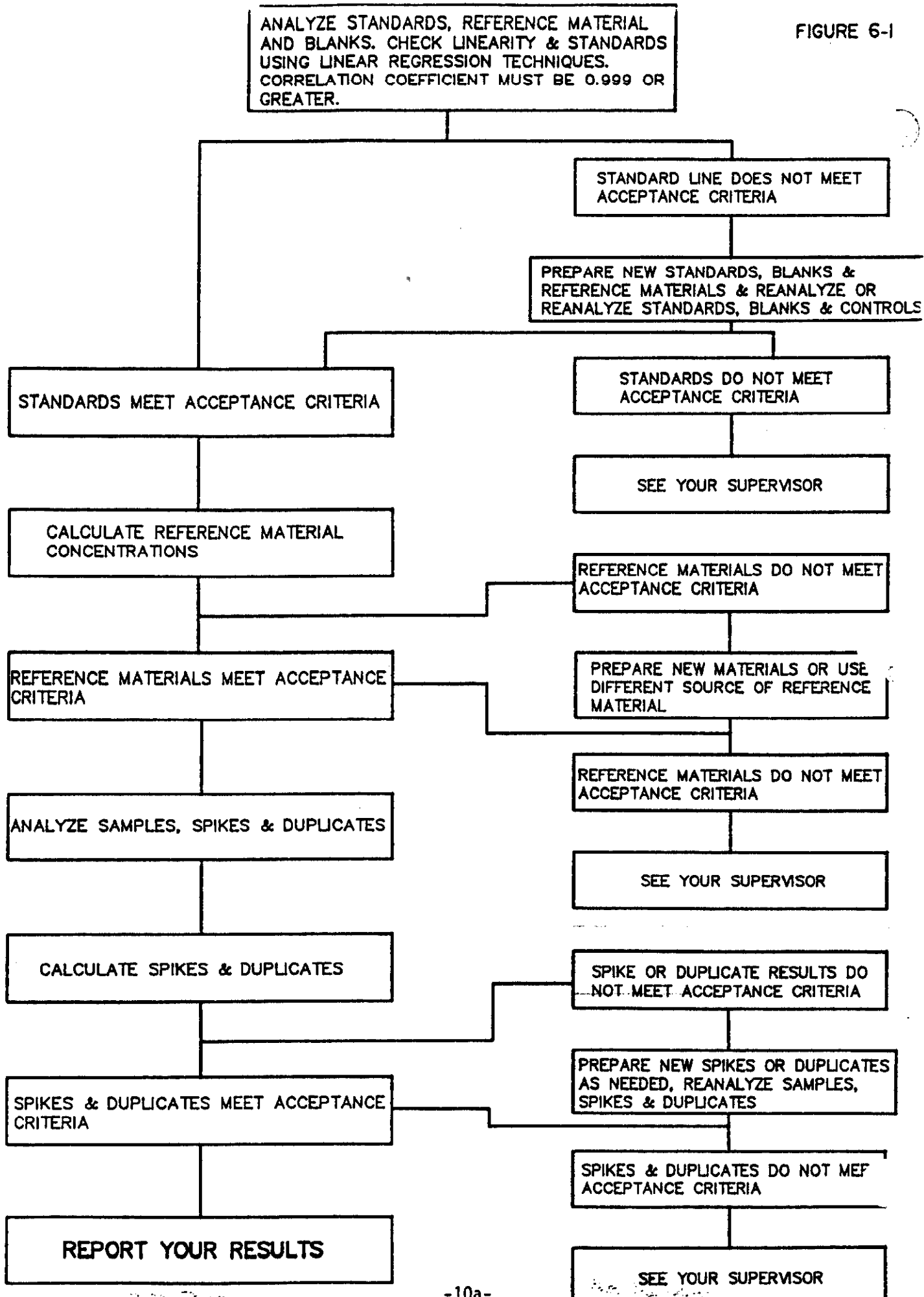
$$\text{Critical Difference} = (X_1 - X_2) \times 3.27$$

For accuracy, the upper and lower confidence levels are calculated by using the following equation for each range of concentration for any particular analyte.

$$100\% \pm (1.965 \times \text{Standard Deviation})$$

Blind field standards will be analyzed on a project by project basis. Reports of performance on blind field standards will be made to the project file. Any discrepancies (data outliers) shall be identified and the source of error eliminated.

Reports of quality assurance/quality control efforts are made verbally to the management of Chen-Northern upon request. A schedule of these reports has not been devised.



## DEFINITION OF TERMS:

**Range:** The range is the boundary of lowest to highest values to which a statement pertains.

**Rc:** Critical range; the maximum allowable difference between a sample value and its duplicate value.

**LCL:** Lower control limit; the smallest acceptable percent recovery allowed for a spiked sample.

**UCL:** Upper control limit; the highest acceptable percent recovery allowed for a spiked sample.

**Example:** A sample is analyzed for calcium. Values determined were 33.4 and 39.6. Is this within acceptable duplicate criteria?

From the quality control acceptance criteria (Table 1) for the range of 10-50, the maximum allowable difference in calcium duplicate is 5. Since the difference of these values is 6.2, the sample determinations are not acceptable.

**Example:** A sample is analyzed for dissolved arsenic content. This value is 0.011 ppm. The sample is spiked to a total concentration of 0.020 ppm and 0.017 ppm is recovered. Is this acceptable?

In the range 0.010 to 0.050, the lowest percent recovery allowed is 90 percent (on a dissolved determination); the highest percent recovery allowed is 109 percent.

0.017 x 100 is 85 percent recovery and  
0.020 determination does not meet with the acceptance  
criteria of the quality control acceptance criteria.



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**QUALITY CONTROL ACCEPTANCE CRITERIA  
PRECISION & ACCURACY REPORT**

Parameter	-----Precision-----		-----Accuracy-----		Detection Limit, mg/l
	Range Of Concentration mg/l	Range Of Duplicate mg/l	LCL % Recovery	UCL % Recovery	
Acidity	1.0 - 10.0	2.8	0	0	1.0
	10.0 - 100.0	5.9	0	0	
	100.0 - 500.0	20	0	0	
	500.0 - 1000.0	40	0	0	
	1000.0 - 10000.0	50	0	0	
Alkalinity	1.0 - 50.0	3	93	105	1.0
	50.0 - 100.0	10	93	105	
	100.0 - 500.0	13	93	105	
	500.0 - 1000.0	35	93	105	
Aluminum	0.1 - 1.0	0.2	89	110	0.1
	1.0 - 5.0	0.3	89	110	
	5.0 - 10.00	0.6	89	110	
Ammonia as N	0.05 - 0.20	0.05	87	111	0.05
	0.2 - 1.0	0.07	87	111	
	1.0 - 10.0	0.6	87	111	
Antimony	0.05 - 0.50	0.05	85	108	0.05
	0.50 - 5.00	0.10	85	108	
	5.0 - 50.0	0.50	85	108	
Arsenic	0.002 - 0.010	0.002	86	112	0.002
	0.010 - 0.050	0.007	88	110	
	0.050 - 0.100	0.010	89	110	

\* May be difficult to achieve, use other quality indicators as an aid in data evaluation.

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Parameter	-----Precision-----		-----Accuracy-----		Detection Limit, mg/l
	Range Of Concentration mg/l	Range Of Duplicate mg/l	LCL % Recovery	UCL % Recovery	
Barium	0.1 - 1.0	0.1	93	105	0.1
	1.0 - 10.0	0.3	93	105	
Benzene	All	20% of Average Value	80	120	0.001
Beryllium	0.005 - 0.050	0.005	84	105	0.005
	0.050 - 0.100	0.008	84	105	
BOD	1.0 - 10.0	3	0	0	1.0
	10.0 - 25.0	9	0	0	
	25.0 - 100.0	15	0	0	
Boron	0.1 - 1.0	0.1	89	109	0.1
	1.0 - 5.0	0.4	89	109	
	5.0 - 10.0	1.0	89	100	
	10.0 - 100.0	6	89	109	
Cadmium	0.0001 - 0.005	0.0004	81	118	0.0001 0.005
	0.005 - 0.010	0.004	88	105	
	0.010 - 0.050	0.009	88	105	
	0.050 - 0.100	0.015	88	105	
Calcium	0.1 - 2.0	0.3 (Tentative)	89	110	0.1 0.1
	2.0 - 10.0	0.5 (Tentative)	94	110	
	10.0 - 50.0	4	94	105	
	50.0 - 100.0	7	94	105	
	100.0 - 200.0	10	91	105	
	200.0 - 500.0	20	91	105	
	500.0 - 1000.0	40	91	105	

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Parameter	-----Precision-----		-----Accuracy-----		Detection Limit, mg/l
	Range Of Concentration mg/l	Range Of Duplicate mg/l	LCL % Recovery	UCL % Recovery	
COD	1.0 - 10.0	2	71	105	1.0
	10.0 - 100.0	12	69	105	
	100.0 - 500.0	21	70	105	
Chloride	1.0 - 10.0	1	86	111	1.0
	10.0 - 50.0	3.0	94	105	
	50.0 - 100.0	6.0	94	105	
	100.0 - 1000.0	16	94	105	
Chromium	0.002 - 0.02	0.004	90	121	0.002 0.02
	0.02 - 0.20	0.02	91	107	
	0.20 - 1.00	0.10	94	108	
	1.00 - 10.00	0.20	94	110	
Conductivity	0.1 - 1.0	0.1	0	0	0.1
	1.0 - 100.0	4	0	0	
	100.0 - 1000.0	48	0	0	
	1000.0 - 5000.0	160	0	0	
	5000.0 - 10000.0	433	0	0	
Copper	0.001 - 0.02	0.004	92	116	0.01 0.02
	0.02 - 0.20	0.02	89	116	
	0.20 - 0.50	0.04	91	109	
	0.5 - 2.00	0.10			
	2.00 - 10.00	0.2	90	107	

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Parameter	-----Precision-----		-----Accuracy-----		Detection Limit, mg/l
	Range Of Concentration mg/l	Range Of Duplicate mg/l	LCL % Recovery	UCL % Recovery	
Ethylbenzene	All	± 20% of Average	80	120	0.001
Fluoride	0.05 - 0.20	0.04	84	108	0.05
	0.2 - 1.0	0.07	94	103	
	1.0 - 2.0	0.1	93	106	
	2.0 - 5.0	0.3	93	112	
	5.0 - 50.0	1.3	93	112	
Iron	0.05 - 0.5	0.07	92	106	0.05
	0.50 - 1.0	0.1	92	106	0.001
	1.00 - 5.0	0.20	92	106	
	5.00 - 10.0	0.32	92	106	
	10.00 - 50.0	1.0	92	106	
Lead	0.001 - 0.020	0.002	81	118	0.02
	0.02 - 0.10	0.03	82	109	
	0.10 - 1.00	0.08	89	105	
	1.00 - 10.00	0.16	89	105	
Magnesium	0.1 - 2.0	0.3 (Tentative)	89	110	0.1
	2.0 - 10.0	1	93	102	
	10.0 - 50.0	4	93	102	
	50.0 - 100.0	5	93	103	
	100.0 - 500.0	10	94	102	
	500.0 - 1000.0	50	93	102	
Manganese	0.02 - 0.10	0.03	87	107	0.02
	0.10 - 0.50	0.03	92	107	
	0.50 - 1.00	0.05	90	108	
	1.00 - 5.00	0.28	95	107	
	5.00 - 10.00	0.50	90	107	
	10.00 - 50.00	1.0	90	107	

May be difficult to achieve, use precision guide to evaluate out of range spikes.

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Parameter	-----Precision-----		-----Accuracy-----		Detection Limit, mg/l
	Range Of Concentration mg/l	Range Of Duplicate mg/l	LCL % Recovery	UCL % Recovery	
Mercury	0.0002 - 0.0020	0.0003	84	110	0.0002
	0.0020 - 0.006	0.0005	84	110	
	0.0060 - 0.020	0.0010	84	110	
Molybdenum	0.05 - 0.100	0.02	91	104	0.05
	0.10 - 1.00	0.08	92	105	
Nickel	0.02 - 0.10	0.02	86	102	0.02
	0.10 - 1.00	0.05	88	102	
Nitrate as N	0.01 - 0.10	0.02	91	109	0.01
	0.10 - 0.50	0.04	90	106	
	0.50 - 2.00	0.10	90	106	
	2.0 - 10.0	0.3			
Petroleum Hydrocarbons	All	20% of Average Value	0	120	Varies
pH	0.10 - 14.0	0.16	0	0	0.10
Phosphate as P	0.005 - 0.020	0.005	84	112	0.005
	0.020 - 0.10	0.02	84	112	
	0.10 - 0.50	0.09	93	108	
	0.50 - 1.00	0.09	93	108	
Potassium	0.5 - 2.00	0.2	90	105	0.5
	2.00 - 10.0	0.5	94	105	
	10.0 - 50.0	3	94	104	
Selenium	0.002 - 0.010	0.002	*89	*110	0.002
	0.010 - 0.050	0.004	89	110	
	0.050 - 0.100	0.010	88	110	

\* May be difficult to achieve, use other quality indicators as an aid in data evaluation.

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Parameter	-----Precision-----		-----Accuracy-----		Detection Limit, mg/l
	Range Of Concentration mg/l	Range Of Duplicate mg/l	LCL % Recovery	UCL % Recovery	
Silver	0.0001 - 0.0050	0.0002	88	118	0.0001 0.02
	0.0050 - 0.010	0.0006	88	118	
	0.01 - 0.10	0.02	85	115	
	0.10 - 1.00	0.08	90	108	
Sodium (Tentative)	0.1 - 2.0	0.3	89	110	0.1
	2.0 - 10.0	2	90	105	
	10.0 - 100.0	5	90	105	
	100.0 - 500.0	15	91	105	
	500.0 - 1000.0	50	91	105	
Sulfate	1.0 - 50.0	6	89	111	1.0
	50.0 - 100.0	12	92	109	
	100.0 - 500.0	33	91	109	
	500.0 - 1000.0	63	91	109	
	1000.0 - 5000.0	90	93	110	
Dissolved Solids	1.0 - 50.0	5	0	0	1.0
	50.0 - 200.0	16	0	0	
	200.0 - 500.0	20	0	0	
	500.0 - 1000.0	38	0	0	
	1000.0 - 5000.0	129	0	0	
	5000.0 - 10000.0	375	0	0	
Suspended Solids	1.0 - 10.0	2	0	0	1.0
	10.0 - 50.0	7	0	0	
	50.0 - 100.0	14	0	0	
	100.0 - 500.0	25	0	0	
	500.0 - 1000.0	50	0	0	
	1000.0 - 10000.0	95% of Average Value			
Gene	All	20% of Average Value	80	120	0.001

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Parameter	-----Precision-----		-----Accuracy-----		Detection Limit, mg/l
	Range Of Concentration mg/l	Range Of Duplicate mg/l	LCL % Recovery	UCL % Recovery	
Vanadium	0.20 - 1.0	0.1	83	101	0.2
Volatile Organics	All	20% of Average Value	80	120	Varies
Xylenes	All	20% of Average Value	80	120	0.001
Zinc	0.02 - 0.10	0.02	88	105	0.02
	0.10 - 0.5	0.004	90	105	
	0.5 - 2.0	0.10	90	105	
	2.00 - 10.00	0.21	90	105	

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## **7.0 CALIBRATION AND PREVENTIVE MAINTENANCE**

Test instruments are calibrated on regular intervals recommended by the manufacturer and as required by ASTM, EPA or other methods. Calibration of all equipment used and documentation of the calibration will be performed by individuals assigned by the laboratory manager or by an independent calibration firm.

Equipment calibrated includes any equipment to be used for analytical testing. The standards, whether reagent or apparatus, used for calibration of equipment are calibrated against a standard traceable to NBS or other recognized physical or chemical constants.

Calibration procedures used are specified by the manufacturer, regulatory agencies, or method. The procedure provides the specific instructions in a step-by-step detail for obtaining and documenting the results. The data are kept on file in the chemical laboratory with the data generated using that instrument calibration. A chronological list of calibration due dates is maintained in a central filing system to maintain proper calibration intervals.

## **8.0 PROCUREMENT CONTROL AND REAGENT QUALITY**

Materials, reagents, and chemicals are received, labeled, stored and issued at Chen-Northern's chemical laboratory according to technical and quality requirements.

Quality standards apply to the vendor supplying the laboratory with analytical materials. Containers, materials or reagents and their packing slips must be marked with the vendor's name and address, the name of the material, the vendor's lot number, quantity and expiration date. Materials received must meet the most recent specifications and the materials must be properly identified.

Purchase orders, packing list certifications, and receiving papers are retained in a central file and are used as a quality control check on the materials received.

The control of incoming materials and reagents is handled by the individual who placed the order. Upon receipt of the supplies, the ordering information is compared with the receiving information. If a discrepancy is found that may affect the quality of the product, the materials are returned. If accepted, the individual ordering the material places the date on the label of the reagent or material, along with the date when the material is no longer capable of peak performance (shelf life). The laboratory manager determines the shelf life or, for certain chemicals, a shelf life is provided by the manufacturer. A first-in first-out usage is maintained by the users. The laboratory manager surveys the store room monthly and disposes of any materials, chemicals or reagents approaching the shelf life expiration date and reorders the chemical.

All organic and inorganic chemicals used in the laboratory for the preparation of primary standards and other solutions conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society. If other grades are used, it first must be ascertained that the chemical is of sufficiently high

purity to permit its use without lessening the accuracy of the analysis. Reagents and solvents are stored in borosilicate glass bottles, metal or polyethylene containers, whichever is appropriate according to method specifications.

Reagents and solvents sensitive to light or temperature are stored in dark bottles or in a cool dark place. The concentration and composition of many reagents are susceptible to change over a short period of time. All reagents and standards are labeled and dated when prepared to monitor their shelf life. The estimated shelf life for the common reagents used are shown in Table 8-1.

Control or reference samples are analyzed with each set of samples for all analytical procedures to insure that the reagents used have not degraded or become contaminated and the instrument performance is meeting its specifications.

Gases used in the laboratory can be classified to serve one of three functions: fuel, oxidant or carrier.

The following is a list of the types of gases used:

<u>Type</u>	<u>Parameter</u>	<u>Use (function)</u>
Air, zero grade	Organic Analysis	Oxidant
Acetylene, Commercial		
Grade	Metals Analysis	Fuel
Nitrous Oxide	Metals Analysis	Oxidant
Compressor Air -		
Supplied by Compressor	Metals Analysis	Oxidant
Argon	Metals Analysis	Carrier
Nitrogen	Metals Analysis	Carrier
Helium, ultra purity	Organics Analysis	Carrier
Hydrogen, ultra purity	Organics Analysis	Fuel

Most fuels and oxidant gases used for atomic absorption work are of commercial grade. Air supplied by a compressor is passed through a filter to remove any oil, water and trace metals from the line for metals analysis only.

There are three grades of water used in the laboratory. Tap Water; Type II ASTM Reagent Water; and Type I ASTM Reagent Water. A description of the various waters used follows:

1. Tap Water - The tap water used in the laboratory is from the Billings City water supply. Its primary use is for the washing of glassware and containers.
2. Type II ASTM Reagent Water - This water is produced by passing tap water through a Millipore reverse osmosis system and Barnstead polishing cartridges. This water has a greater than 1 megaohm resistance. This water is primarily used for preparing reagents, solutions and standards. It is also used as the final rinse for most of the laboratory glassware and containers.
3. Type I ASTM Reagent Water - This higher quality water is produced by treating the Type II water with a Millipore Super Q four bed recirculating system. This water is available on demand at up to 3 gpm.

The water is checked to determine if each type is meeting the criteria listed in Table 8-2. The resistance is monitored weekly at various laboratory outlets to verify the quality of distilled water.

The type of glassware and its cleanliness are important factors in obtaining accurate analytical results in the laboratory. The glassware used in the laboratory for volumetric and colorimetric analyses are borosilicate Kimax or Pyrex brand glass.

Other glassware meets the Class A specifications of the National Bureau of Standards with reference to capacity and delivery.

The cleaning method for the glassware is dependent upon the substances that are to be removed and the use of the glassware. Water-soluble substances are removed with tap water and demineralized or distilled water. Other substances more difficult to remove require the use of an acid cleaning solution as well. Special cleaning techniques are necessary for glassware used for certain laboratory analysis.

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TABLE 8-1

## REAGENT SHELF LIFE LIST

<u>Parameter</u>	<u>Reagent</u>	<u>Container Type</u>	<u>Storage or Shelf Life</u>	<u>Remarks</u>
Asbestos Fibers	Mounting Solution	Balsam Bottle	3 Months	None
Asbestos Identification	Dispersion Liquids	Amber Glass	1 Year	None
Ammonia	Stock Ammonia	Glass	1 Month	
Ammonia	Mixed Indicator	Glass	3 Months	
Ammonia	Nessler Reagent	Dark Glass with rubber stopper	6 Months	
Ammonia	Borate Buffer Solution	Glass or Plastic	3 Months	
Ammonia	Sodium Hydroxide	Plastic	3 Months	
Ammonia	Boric Acid Solution	Glass	1 Month	
Ammonia	H <sub>2</sub> SO <sub>4</sub> 0.02 N	Glass or Plastic	3 Months	standardize daily
Boron	Stock Boron Solution	Plastic	3 Months	
Bromide	Acetate Buffer Solution	Glass	3 Months	Store at 4°C
Bromide	Chloramine-T Solution	Brown Glass	3 Months	
Bromide	Phenol Red Indicator	Glass or Plastic	3 Months	
Bromide	Sodium Thiosulfate	Glass or Plastic	3 Months	
		standardize daily		
Bromide	Stock Bromide Solution	Glass or Plastic	3 Months	
Chlorides	Standard Sodium Chloride	Plastic	3 Months	
Chlorides	Nitric Acid 1N	Glass or Plastic	3 Months	
Chlorides	Sodium Hydroxide 1N	Plastic	3 Months	
Low Chlorides	Indicator Reagent	Brown Glass	1 Month	Store at 4°C
Low Chlorides	Standard Mercuric Nitrate	Brown Glass	3 Months	standardize daily

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TABLE 8-1 (cont.)  
REAGENT SHELF LIFE LIST

<u>Parameter</u>	<u>Reagent</u>	<u>Container Type</u>	<u>Storage or Shelf Life</u>	<u>Remarks</u>
High Chlorides	Mixed Indicator Reagent	Glass	3 Months	
High Chlorides	Strong Standard Mercuric Nitrate	Brown Glass	3 Months - standardize daily	
Chlorine Residual				
Iodometric	Standard Sodium Thiosulfate	Glass or Plastic	3 Months	Standardize daily
Iodometric	Standard Iodine Solution	All Glass Dark	3 Months	Standardize daily
Iodometric	Starch Solution	Glass or Plastic	3 Months	
Chromium, Hex	APDC Solution	Amber Glass	3 Months or Discoloration	
Cyanide	Sodium Hydroxide Solution	Plastic	3 Months	
Cyanide	Cuprous Chloride Reagent	Glass or Plastic with Copper Wire in Bottle		
Cyanide	Sodium Dihydrogen Phosphate	Plastic	3 Months	Store at 4°C
Cyanide	Stock Cyanide Solution	Glass or Plastic	3 Months	
Cyanide	Standard Silver Nitrate	Glass	3 Months	
Cyanide	Rhodanine Indicator	Glass	3 Months	
Cyanide	Chloramine T	Glass or Plastic	1 Week	Store at 4°C
Cyanide	Pyridine Barbituric Acid	Dark Glass	1 Month	
Cyanide	Stock Cyanide Solution	Plastic or Glass	6 Months	
Fluoride	Standard Fluoride Solution	Glass	3 Months	
Fluoride	Stock Fluoride Solution	Glass	3 Months	
Fluoride	TISAB Reagent	Glass	1 Month	
Hardness	Buffer Solution	Plastic or Pyrex Glass	1 Month	NOTE: Be sure to have bottle stop- pered tightly to prevent loss of $\text{HN}_3$

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TABLE 8-1 (cont.)

## REAGENT SHELF LIFE LIST

<u>Parameter</u>	<u>Reagent</u>	<u>Container Type</u>	<u>Storage or Shelf Life</u>	<u>Remarks</u>
Hardness	EDTA Titrant 0.01M	Plastic	3 Months	Standardize daily
Hardness	Standard Calcium Solution	Glass or Plastic	3 Months	
Nitrogen -				
Kjeldahl & Ammonia	Alkaline Phenol	Plastic or Glass	3 Months	
Kjeldahl & Ammonia	Sodium Hypochlorite	Plastic	1 Month	
Kjeldahl & Ammonia	Disgestant Mixture	Glass	3 Months	
Kjeldahl & Ammonia	Stock Ammonium Chloride	Glass or Plastic	3 Months	
Kjeldahl & Ammonia	Digestion Reagent	Glass	3 Months	
Kjeldahl & Ammonia	Sodium Hydroxide/Thiosulfate	Plastic	3 Months	
Kjeldahl & Ammonia	Sodium Thiosulfate	Glass	1 Month	
Kjeldahl & Ammonia	Mix Indicator	Glass Stoppered	1 Month	
Kjeldahl & Ammonia	Indicator Boric Acid	Glass	1 Month	
Kjeldahl & Ammonia	0.02 N Sulfuric Acid	Glass	3 Months	
Nitrogen -				
Nitrate-Nitrite	Combined Color Reagent	Glass	3 Months	
Nitrate-Nitrite	Ammonium Chloride	Plastic or Glass	3 Months	
Nitrate-Nitrite	Stock Nitrate Solution	Plastic or Glass	3 Months	
Nitrate-Nitrite	Standard Copper Sulfate	Glass	3 Months	
Nitrate-Nitrite	EDTA	Glass or Plastic	3 Months	
Nitrate-Nitrite	EDTA	Glass or Plastic	3 Months	
Nitrate-Nitrite	Sulfanilic Acid Reagent	Glass or Plastic	3 Months	
Nitrate-Nitrite	Naphthylamine Reagent	Glass	3 Months	
Nitrate-Nitrite	Sodium Acetate Buffer			
	Solution	Glass or Plastic	3 Months	
Nitrate-Nitrite	Stock Nitrite Solution	Glass or Plastic	1 Month	
Petroleum Hydrocarbons	Standard Solution	Glass	4 Weeks	



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TABLE 8-1 (cont.)

## REAGENT SHELF LIFE LIST

<u>Parameter</u>	<u>Reagent</u>	<u>Container Type</u>	<u>Storage or Shelf Life</u>	<u>Remarks</u>
Phenol	Aminoantipyrine and Potassium Ferricyanide	Glass	1 Day	
Phenol	Ammonium Chloride	Glass or Plastic	3 Months	
Phenol	Ammonium Hydroxide	Glass	3 Months	
Phenol	Bromate Bromide Solution	Glass	3 Months	
Phenol	Starch Solution	Glass or Plastic	3 Months	
Phenol	Potassium Ferricyanide	Glass or Plastic	1 Week	
Phenol	Stock Phenol Solution	Glass	1 Month	
Phosphate -				
Ascorbic Acid	5N Sulfuric Acid	Glass	3 Months	
Ascorbic Acid	Antimony Potassium Tartrate	Dark Glass and Stoppers	3 Months	Store at 4°C
Ascorbic Acid	Ammonium Molybdate	Plastic	3 Months	Store at 4°C
Ascorbic Acid	Ascorbic Acid	Glass	1 Week	Store at 4°C
Ascorbic Acid	11 N Sulfuric Acid	Glass	3 Months	
Ascorbic Acid	Stock Phosphate	Glass	3 Months	Store at 4°C
Silica	Sulfuric Acid 1N	Plastic	3 Months	
Silica	Hydrochloric Acid 1+1	Plastic	3 Months	
Silica	Ammonium Molybdate Reagent	Plastic	3 Months	
Silica	Oxalic Acid	Plastic	3 Months	
Silica	Stock Silica Solution	Plastic	3 Months	
Sulfate	Barium Chloride 10% Reagent	Glass or Plastic	6 Months	
	Standard Sulfate Solution	Glass or Plastic	3 Months	

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TABLE 8-1 (cont.)

### **REAGENT SHELF LIFE LIST**

<u>Parameter</u>	<u>Reagent</u>	<u>Container Type</u>	<u>Storage or Shelf Life</u>	<u>Remarks</u>
Sulfide	Hydrochloric Acid 6N	Glass	3 Months	
Sulfide	Standard Iodine Solution	Glass	3 Months	
Sulfide	Standard Sodium Thiosulfate	Glass or Plastic	3 Months	Standardize daily
Sulfide	Starch Solution	Glass or Plastic	3 Months	Standardize daily
Sulfite	Sulfuric Acid 1+1	Glass or Plastic	3 Months	
Sulfite	Starch Indicator	Glass or Plastic	3 Months	
Sulfite	Standard Potassium Iodide-Iodate	Glass or Plastic	3 Months	Standardize daily
Sulfur	Methyl Orange Rinse Solution	Glass or Plastic	3 Months	
Sulfur	Sodium Carbonate Titrant	Glass or Plastic	3 Months	
Sulfur	Ammonium Hydroxide	Glass or Plastic	3 Months	
Sulfur	Bromine Water	Glass or Plastic	3 Months	
Sulfur	5 N Sodium Hydroxide	Plastic	3 Months	
Sulfur	3 N Hydrochloric Acid	Glass or Plastic	3 Months	
Sulfur	10% Barium Chloride Solution	Glass or Plastic	3 Months	
Volatile Organics	Standard Solution	Glass	4 Weeks	

TABLE 8-2  
WATER QUALITY CRITERIA

	Type I	Type II
Total matter, max. mg/litre	0.1	0.1
Electrical conductivity, max. umho/cm at 298 K (25°C)	0.06	1.0
Electrical resistivity, min. M cm at 298 K (25°C)	16.67	1.0
pH at 298 K (25°C)	A	A
Minimum color retention time of potassium permanganate, minutes	60	60
Maximum soluble silica	not detectable	not detectable

-----  
Microbiological classification<sup>B</sup>

<sup>A</sup>The measurement of pH in Type I and II reagent waters is meaningless and has been eliminated from the procedure, since electrodes used in this test contaminate the water.

<sup>B</sup>When bacterial levels need to be controlled, reagent grade types should be further classified as follows:

	Type A	Type B	Type C
Maximum total bacteria count	0/ml	10/ml	100/ml

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## 9.0 TRAINING

All Chen-Northern employees are trained to perform the tasks assigned to them. This consists of on-the-job training. Short courses and specialty conferences are included when appropriate. All technical personnel are required to attend an indoctrination program administered by the laboratory manager. The indoctrination program covers employment requirements, policies, procedures, and objectives of Chen-Northern and stresses the aspects of its quality assurance program.

All new employees are considered probationary for a period of six months from the time of employment. The training program for new personnel is administered by the laboratory manager and consists of on-the-job training directed by the laboratory supervisor. The trainee is required to read the appropriate standards, methods or standard operating procedures and to become familiar with the equipment and measurements used in testing. The trainee observes an experienced operator perform the tests and then performs the test under the direct supervision of the operator.

Before any test is performed by a trainee without direct supervision, the laboratory supervisor observes the trainee performing the test and then initials the trainee's "Personnel Proficiency Check Sheet" (Figure 9-1). Maintenance of the proficiency check sheet system is the responsibility of the laboratory manager. The personnel progress check sheets are filed in the chemical laboratory. The progress check sheets are examined annually to determine employee growth, supervisory efficiency, compatibility of job objectives and overall job knowledge, including a review of wage/salary to determine if compensation is compatible with job progress, duties and responsibilities.

It is Chen-Northern's policy to provide for the continuing training and development of its technical personnel. The program is administered by the laboratory manager and provides for the following:

- a) Selected external programs for attendance by key personnel. Program selection and attendance must have approval of both the laboratory manager and the Regional Vice-President.
- b) Selected special training for specialized positions or for newly created positions may include assistance for attendance at accredited educational institutions. The laboratory manager has the responsibility for selecting and recommending participation in these programs.
- c) The laboratory manager will review and evaluate the development programs once a year regarding the effectiveness of the programs and changes to be made.

FIGURE 9-1  
PERSONNEL PROFICIENCY CHECK SHEET

Employee Name:

Date of Employment:

Analyses	Equipment Operation	Date	Initial of Lab Manager
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Record of Continuing Education:

<u>Seminars, Classes, etc.</u>	<u>Date</u>	<u>CEU's</u>
--------------------------------	-------------	--------------

## 10. SAFETY

Appropriate safety techniques and procedures are required with the continual expansion and sophistication of techniques, chemicals and equipment used in an environmental laboratory. It is never assumed that personnel at any level of work have adequate information about laboratory safety. For this reason, the need for a training program is recognized to insure a safe laboratory environment.

This program involves the availability of proper safety equipment and adequate personnel training. The following is a list of the safety equipment located in the laboratory:

- ♦ Emergency shower
- ♦ Gas mask
- ♦ Eye wash fountain
- ♦ Eye wash solution
- ♦ Fire extinguishers
- ♦ Emergency blanket
- ♦ First aid kit
- ♦ Thermal gloves
- ♦ Safety glasses
- ♦ Laboratory coats
- ♦ Fume exhaust hoods

Fume hoods are provided in the laboratory for safe use of gaseous or toxic reagents. The reagents are stored in proper containers at ambient temperature in specifically designated areas of the laboratory. These areas are segregated to avoid contact of incompatible hazardous materials. Proper grounding of electrical equipment in the laboratory is inspected and monitored as a part of the routine preventative maintenance program.



## 11.0 ANALYTICAL METHODS

The analytical methods used by Chen-Northern for the analysis of water and wastes have been documented by the U.S. Environmental Protection Agency as approved methodologies, under the National Pollutant Discharge Elimination System (NPDES) Permit Program or the Safe Drinking Water Act. Most of the procedures used by Chen-Northern are from the following sources: Standard Methods for the Examination of Water and Wastewater; Methods for Chemical Analysis of Water and Wastes, U.S. EPA; ASTM Water, EPA SW-846, "Test Method for Evaluating Solid Wastes."

Methods for the analysis of materials for asbestos are from NIOSH Manual of Analytical Methods and EPA's Interim Method for the Determination of Asbestos in Bulk Insulation Samples, EPA 600/4-82-020. Methods used for the analysis of soils and overburden are from the USDA Handbook 60, Diagnosis and Improvement of Saline and Alkali Soils and American Society of Agronomy Monograph 9, Methods of Soil Analysis and others.

Methodologies employed are documented in Table 11-1 for specific analyses. These methods specify at a minimum:

- ♦ appropriate instrumentation and equipment
- ♦ instrument calibration
- ♦ reagent quality and concentration
- ♦ reagent standardization
- ♦ analytical procedure
- ♦ method of calculation of results

These methods generally describe the conversion of raw data (absorbances, weights, volumes, etc.) to concentration units. As a matter of standard practice, concentrations as a result of gravimetric analysis are determined by calculation using weights and

volumes measured in the test. Concentrations, as a result of spectrophotometric analysis are calculated by comparing absorbance units of standards and samples using linear regression equations.

TABLE 11-1

## ANALYTICAL METHODOLOGIES

Parameter	Methodology*
Acidity	Titrimetric, EPA Method 305.1
Alkalinity	Titrimetric, EPA Method 310.1 Colorimetric, Automated Methyl Orange, EPA Method 310.2
Aluminum	Direct Aspiration, ICP AA, Furnace, EPA Method 202.2
Antimony	Direct Aspiration, ICP Furnace, AA, EPA Method 204.1
Arsenic	Hydride, AA, EPA 206.3 Furnace, AA, EPA 206.2
Barium	Direct Aspiration, ICP Direct Aspiration, AA, EPA Method 208.1
Beryllium	Direct Aspiration, ICP Direct Aspiration, AA, EPA Method 210.1 Furnace, AA, EPA Method 208.2
Benzene	Gas Chromatograph EPA 602, 8020
Boron	Direct Aspiration, ICP
Cadmium	Direct Aspiration, ICP Direct Aspiration, AA, EPA Method 213.1 Furnace, AA, EPA Method 213.2
Calcium	Direct Aspiration, ICP Direct Aspiration, AA, EPA Method 215.1 Titrimetric, EDTA, EPA Method 215.2
Chloride	Titrimetric, Mercuric Nitrate, EPA Method 325.3 Colorimetric, Automated $\text{Fe}_3(\text{CN})_6$ , AAIL, EPA Method 325.2
Chlorophyll-a	Spectrophotometric, SM1002G

\*See Index to Abbreviations (last page)

TABLE 11-1 (cont.)

## ANALYTICAL METHODOLOGIES

Parameter	Methodology*
Chromium	Direct Aspiration, ICP Furnace, AA, EPA Method 218.2
Cobalt	Direct Aspiration, ICP Furnace, AA, EPA 219.2
Color	Colorimetric, Platinum Cobalt, EPA 110.2 Spectrophotometric, EPA 110.3
Copper	Direct Aspiration, ICP Furnace, AA, EPA 220.2
Cyanide:	
a) Photometric Determination of Simple Cyanide	Spectrophotometric, EPA 335.1
b) Total Cyanide after Distillation	Spectrophotometric, EPA 335.2
Ethyl Benzene	Gas Chromatograph EPA 602, 8020
Fluoride	Potentiometric, Ion Selective Electrode, EPA 340.2
Hardness	Colorimetric, Automated EDTA, EPA 130.1 Titrimetric, EDTA, 130.2
Iron	Direct Aspiration, ICAP Furnace, AA, EPA 236.2
Lead	Direct Aspiration, ICAP Furnace, AA, EPA 239.2
Magnesium	Direct Aspiration, ICAP Direct Aspiration, AA, EPA 242.1
Manganese	Direct Aspiration, ICAP Furnace, AA, EPA 243.2
Mercury	Cold Vapor, Manual, EPA 245.1

TABLE 11-1 (cont.)

## ANALYTICAL METHODOLOGIES

Parameter	Methodology*
Molybdenum	Direct Aspiration, ICAP Furnace, AA, EPA 246.2
Nickel	Direct Aspiration, ICAP Furnace, AA, EPA 249.2
Nitrogen:	
a) Ammonia	Colorimetric, Titrimetric, Distillation Procedure, EPA 350.2
b) Kjeldahl	Colorimetric, Titrimetric, Potentiometric, EPA 351.3
c) Nitrate	Colorimetric, Automated Cadmium Reduction, EPA 353.2
d) Nitrite	Colorimetric, EPA 353.2
e) Organic	Kjeldahl minus Ammonia (see above)
Oil and Grease	Gravimetric, Separatory Funnel Extraction, EPA 413.1
Oxygen:	
a) Biochemical Demand	BOD - 5 Day, EPA 405.1
b) Chemical Demand	Titrimetric, Low, Mid, High Level, EPA 410.1-.3
c) Dissolved	
Petroleum Hydrocarbons	Infrared 418.1, Gas Chromatograph 8015 or California Method
pH	Electrometric, EPA 150.1
Phenols:	
a) Direct Photometry After Distillation	Spectrophotometric, Manual 4 - AAP, EPA 420.1
b) Chloroform Extrac- tion after Distil- lation	Spectrophotometric, Manual 4 - AAP, EPA 420.1
Phosphorus:	
a) Ortho	ALL FORMS - Colorimetric, Automated Ascorbic Acid, EPA 365.1
b) Total	Colorimetric, Ascorbic Acid, Single Reagent, EPA 365.2

TABLE 11-1 (cont.)

## ANALYTICAL METHODOLOGIES

Parameter	Methodology*
Potassium	Flame Photometric Method, SM 322B AA, Direct Aspiration, EPA 258.1
Selenium	AA, Furnace, EPA Method 270.2 AA, Hydride, EPA Method 270.3
Silicon as SiO <sub>2</sub>	Direct Aspiration, ICP Colorimetric, EPA 370.1
Silver	Direct Aspiration, ICP AA, Furnace, EPA 272.2
Sodium	Flame Photometric Method, SM 325B AA, Direct Aspiration, EPA 273.1
Residue:	
Filtrable (TDS)	Gravimetric at 180°C, EPA 160.1
Suspended	Gravimetric at 103-105°C, EPA 160.2
Settleable	Volumetric, Imhoff Cone, EPA 160.5
Volatile	Gravimetric, Ignition at 550°, EPA 160.4
Conductivity	Specific Conductance, EPA 120.1
Sulfate	Gravimetric, EPA 375.3 Colorimetric, Automated Methyl Thymol Blue, EPA 375.2
Sulfide	Titrimetric Iodine, EPA 376.1
Thallium	AA, Furnace, EPA 279.2
Toluene	Gas Chromatograph EPA 602,8020
Turbidity	Nephelometric, EPA 180.1
Vanadium	Direct Aspiration, ICP AA, Furnace, EPA 286.1
Volatile Organics (Regulated for Drinking Water)	Method 502.2 GC/Hall Detector with Purge and Trap
Xylenes	Gas Chromatograph EPA 602, 8020
Zinc	Direct Aspiration, ICP AA, Furnace, EPA 289.2

TABLE 11-1 (cont.)

## ANALYTICAL METHODOLOGIES

Parameter	Methodology*
Digestion Technique for Total Recoverable Metals	Metal S-6, EPA 4.14
Moisture	ASTM D3173
Ash	ASTM D3174
Total Sulfur	ASTM D3177
Pyritic Sulfur	ASTM D2492
Sulfate Sulfur	ASTM D2492
Heat of Combustion	ASTM D3286
Major & Minor Elements	ASTM D3682
Trace Elements	ASTM D3683
pH	ASA 60-3
Electrical Conductivity	ASA 62-2
Sodium Absorption Ratio	USDA, Page 72
Calcium	USDA 3a, then ICP
Magnesium	USDA 3a, then ICP
Sodium	USDA 3a, then ICP
Saturation	USDA 27
Particle Size	USDA 41
Texture	USDA 41
Carbonate	ASA 91-4

TABLE 11-1 (cont.)

## ANALYTICAL METHODOLOGIES

Parameter	Methodology*
Selenium	ASA 80-2
Boron	ASA 74-3, 74-4
Nitrate	ASA 84-5
Organic Matter	ASA 92-3
Molybdenum	ASA 74-1
Copper	DPTA, ICP
Lead	DPTA, ICP
Arsenic	EPA 206.3
Potassium	ASA 71-2, 71-4, USDA 11a
Phosphorus	ASA 73-4
Ammonia	ASA 88-3
Exchangeable Cations	USDA 18, 19
Asbestos Content	EPA Interim Method 600/M4-82-020
Asbestos Fibers	NIOSH P&CAM 239

## Abbreviations used in above listings:

- EPA: Methods for Chemical Analysis of Water and Wastes  
EPA 600/4-79-020
- SM: Standard Methods for the Examination of Water and Wastewater  
16th Edition, APHA-AWWA-WPCF, 1983
- ICP: Inductively Coupled Plasma-Atomic Emission Spectrometric  
Method for Trace Element Analysis of Water and Wastes,  
Method 200.7, U.S. Environmental Protection Agency,  
Environmental Monitoring and Support Laboratory, Cincinnati,  
Ohio, 45268, November 1980.
- ASTM: American Society of Testing Materials



USDA:           USDA Handbook 60, Diagnosis and Improvement of Saline  
and Alkali Soils

ASA:           American Society of Agronomy Monograph 9, Methods of  
Soil Analysis

NIOSH:         NIOSH "Manual of Analytical Methods"

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## 12.0 SIGNIFICANT NUMBERS

The primary objective is to report all results in such a way that they can be interpreted properly with reference to the accuracy of the test.

The attached significant place sheet specifically lists the parameters and the number of significant places that a result should be reported for a specific range. Also included is a minimum detection limit for that parameter. It should be noted, however, that this detection limit is the routine minimum detection limit and often a lower detection limit can be reached by altering the test in some way.

To use the significant place sheet:

1. Look up the parameter
2. Check to see that the result is not below the minimal detection limits; if it is, the result should be reported as less than this limit.
3. Find the range that the result lies within.
4. Report the result to the number of significant places as indicated by X's.

**Rounding Off Number:** The following rules should be used for rounding off numbers to the correct significant places:

	<u>Examples</u>
If number ends in 1-4, round off to smaller number:	364 - 360
If number ends in 6-9, round off to larger number:	487 - 490
If number ends in 5, round off to even number:	365 - 360 375 - 380

TABLE 12-1  
REPORT OF RESULTS  
TABLE OF SIGNIFICANT FIGURES

Parameter	Minimum Detection Limit		Range	Significant Figures
Asbestos Fibers	Sample Volume Dependent		0.001-0.010	0.00X
			0.010-0.100	0.0XX
			0.10-1.0	0.XX
			1.0-10	X.X
Asbestos Identification	1	%	1-10	X
			10-100	XX
Acidity	1	mg/l	1-10	X
			10-100	XX
			100-1000	XXX
			1,000-10,000	XXX0
Alkalinity	1	mg/l	1-10	X
			10-100	XX
			100-1000	XXX
			1,000-10,000	XXX0
Boron	0.1	mg/l	0.1-1.0	0.X
			1.0-10	X.X
			10-100	XX
Calcium	1	mg/l	1-10	X
			10-100	XX
			100-1000	XXX
			1,000-10,000	XXX0
Calcium Hardness	3	mg/l	3-10	X
			10-100	XX
			100-1000	XXX
			1,000-10,000	XXX0
Chloride	1	mg/l	1-10	X
			10-100	XX
			100-1000	XXX
			1,000-10,000	XXX0
Chlorine Residual	0.1	mg/l	0.1-1.0	0.X
			1.0-10	X.X
			10-100	XX
			100-1000	XX0
Color	5 color units		5-100	XX
			100-1000	XX0
			1,000-10,000	XX00

TABLE 12-1 (cont.)

REPORT OF RESULTS  
TABLE OF SIGNIFICANT FIGURES

Parameter	Minimum Detection Limit		Range	Significant Figures
Cyanide	0.01	mg/l	0.01-0.1 0.1-1.0 1.0-10 10-100 100-1000	0.0X 0.XX X.X XX XX0
Fluoride	0.10	mg/l	0.1-1.0 1.0-10 10-100 100-1000	0.XX X.X XX XX0
-----				
METALS				
Barium (ICP)	0.10	mg/l	0.1-1.0 1.0-10 10-100	0.X X.XX XX.X
Beryllium (ICP)	0.005	mg/l	0.005-0.010 0.01-0.1 0.1-1.0 1.0-10 10-100	0.00X 0.0XX 0.XX X.X XX
Cadmium (ICP)	0.01	mg/l	0.01-0.1 0.1-1.0 1.0-10 10-100 100-1000	0.0X 0.XX X.X XX XX0
Cadmium (flameless)	0.001	mg/l	0.001-0.010 0.01-0.1 0.1-1.0	0.00X 0.0XX 0.XXX
Cobalt (ICP)	0.02	mg/l	0.02-0.10 0.10-1.0 1.0-10 10-100	0.0X 0.XX X.X XX
Cobalt (flameless)	0.001	mg/l	0.001-0.010 0.010-0.10 0.10-1.0	.00X .0XX .XXX

TABLE 12-1 (cont.)

REPORT OF RESULTS  
TABLE OF SIGNIFICANT FIGURES

Parameter	Minimum Detection Limit		Range	Significant Figures
Chromium (ICP)	0.02	mg/l	0.02-0.10 0.10-1.0 1.0-10 10-100	0.0X 0.XX X.XX XX
Chromium (flameless)	0.002	mg/l	0.002-0.010 0.010-0.10 0.10-1.0	0.00X 0.0XX 0.XXX
Hexavalent Chromium	0.02	mg/l	0.02-0.10 0.10-1.0 1.0-10 10-100	0.0X 0.XX X.X XX
Grease or Oil	1	mg/l	1-10 10-100 100-1000 1,000-10,000	X XX XX0 XX00
Hardness, Total	2	mg/l	2-10 10-100 100-1000 1,000-10,000	X XX XXX XXX0
Iodide	0.01	mg/l	0.01-0.1 0.1-1.0 1.0-10 10-100	0.0X 0.X X.X XX
Silver (ICP)	0.04	mg/l	0.04-0.1 0.10-1.0 1.0-10 10-100	0.0X 0.XX X.X XX
Silver (flameless)	0.001	mg/l	0.001-0.010 0.010-0.10 0.10-1.0	0.00X 0.0XX 0.XXX
Aluminum (ICP)	0.1	mg/l	0.1-1 1-10 10-100 100-1000	0.X X XX XX0

TABLE 12-1 (cont.)

REPORT OF RESULTS  
TABLE OF SIGNIFICANT FIGURES

Parameter	Minimum Detection Limit	Range	Significant Figures
Arsenic (flameless)	0.001 mg/l	0.001-0.010	.00X
		0.010-0.10	.0XX
		0.10-1.0	.XXX
Barium (ICP)	0.1 mg/l	0.1-1.0	0.XX
		1.0-10	X.X
		10-100	XX
		100-1000	XX0
Copper (ICP)	0.02 mg/l	0.02-0.10	0.0X
		0.10-1.0	0.XX
		1.0-10	X.XX
		10-100	XX
		100-1000	XX0
Iron (ICP)	0.05 mg/l	0.05-0.10	0.0X
		0.10-1.0	0.XX
		1.0-10	X.X
		10-100	XX
		100-1000	XX0
Mercury (flameless)	0.5 ug/l	0.5-1.0	0.X
		1.0-10	X.X
		10-100	XX
		100-1000	XX0
Potassium (flame)	1 mg/l	1-10	X
		10-100	XX
		100-1000	XX0
Lithium (ICP)	0.01 mg/l	0.02-0.10	0.0X
		0.10-1.0	0.XX
		1.0-10	X.X
		10-100	XX
		100-1000	XX0
Silica (ICP)	1 mg/l	1-10	X
		10-100	XX
		100-1000	XX0
Magnesium (flame)	1 mg/l	1.0-10	X
		10-100	XX
		100-1000	XXX

TABLE 12-1 (cont.)

REPORT OF RESULTS  
TABLE OF SIGNIFICANT FIGURES

Parameter	Minimum Detection Limit	Range	Significant Figures
Manganese (ICP)	0.02 mg/l	0.02-0.10 0.10-1.0 1.0-10 10-100 100-1000	0.0X 0.XX X.X XX XX0
Molybdenum (ICP)	0.05 mg/l	0.05-0.10 0.1-1 1-10 10-100 100-1000	0.0X 0.XX X.XX XX XX0
Molybdenum	0.001 mg/l	0.001-0.010 0.010-0.10 0.10-1.0	.00X .0XX .XX0
Sodium (flame)	1 mg/l	1-10 10-100 100-1000	X XX XXX
Nickel (ICP)	0.02 mg/l	0.02-0.10 0.10-1.0 1.0-10 10-100 100-1000	0.0X 0.XX X.X XX XX0
Lead (ICP)	0.1 mg/l	0.1-1.0 1.0-10 10-100 100-1000	0.X X.X XX XX0
Lead (flameless)	0.005 mg/l	0.005-0.010 0.010-0.10 0.10-1.0	.00X .0XX .XXX
Antimony (ICP)	0.05 mg/l	0.05-0.10 0.1-1.0 1.0-10 10-100 100-1000	0.0X 0.XX X.XX XX XX0
Selenium (flameless)	0.001 mg/l	0.001-0.010 0.010-0.10 0.10-1.0	.00X .0XX .XX0



TABLE 12-1 (cont.)

REPORT OF RESULTS  
TABLE OF SIGNIFICANT FIGURES

Parameter	Minimum Detection Limit		Range	Significant Figures
Tin (ICP)	0.02	mg/l	0.02-0.10	.0X
			0.10-1.0	.XX
			1.0-10	X.X
Strontium (ICP)	0.05	mg/l	0.05-0.10	0.0X
			0.10-1.0	0.XX
			1.0-10	X.X
			10-100	XX
Titanium (IC))	1	mg/l	2-10	X
			10-100	XX
			100-1000	XX0
Vanadium (ICP)	0.1	mg/l	0.2-1.0	.X
			1.0-10	X.X
			10-100	XX
Zinc (ICP)	0.02	mg/l	0.02-0.10	0.0X
			0.10-1.0	0.XX
			1.0-10	X.X
			10-100	XX
			100-1000	XX0
<u>NITROGEN</u>				
Ammonium (Automated)	0.1	mg/l	0.10-1.0	0.XX
			1.0-10	X.X
			10-100	XX
			100-1000	XX0
Ammonia (Titrimetric)	0.1	mg/l	0.1-1.0	0.X
			1.0-10	X.X
			10-100	XX
			100-1000	XX0
Nitrates	0.05	mg/l	0.05-0.1	0.0X
			0.1-1.0	0.XX
			1.0-10	X.X
			10-100	XX
			100-1000	XX0

TABLE 12-1 (cont.)

REPORT OF RESULTS  
TABLE OF SIGNIFICANT FIGURES

Parameter	Minimum Detection Limit		Range	Significant Figures
Nitrites	0.05	mg/l	0.05-0.1	0.0X
			0.1-1.0	0.XX
			1.0-10	X.X
			10-100	XX
			100-1000	XX0
-----				
Kjeldahl Nitrogen	0.1	mg/l	0.1-1.0	0.X
			1.0-10	X.X
			10-100	XX
			100-1000	XX0
-----				
<u>OXYGEN DEMAND</u>				
BOD <sub>5</sub>	5	mg/l	5-10	X
			10-20	XX
			20-100	XX
			100-1000	XX0
			1,000-10,000	XX00
			10,000-100,000	XX000
-----				
COD (low level)	0.5	mg/l	0.5-1.0	0.X
			1.0-10	X.X
-----				
COD (high level)	5	mg/l	5-10	X
			10-100	XX
			100-1000	XXX
			1,000-10,000	XXX0
-----				
Oil or Grease	1	mg/l	1-10	X
			10-100	XX
			100-1000	XXX
			1,000-10,000	XXX0
-----				
pH	0.1	pH unit	0.1-1.0	0.X
			1.0-10	X.X
			10-14	XX.X
-----				
Phenol	0.005	mg/l	0.005-0.10	.0XX
			0.10-1.0	.XX
			1.0-10	X.X
			10-100	XX
			100-1000	XX0

TABLE 12-1 (cont.)

REPORT OF RESULTS  
TABLE OF SIGNIFICANT FIGURES

Parameter	Minimum Detection Limit	Range	Significant Figures
Organic Nitrogen	0.1 mg/l	0.1-1.0	0.X
		1.0-10	X.X
		10-100	XX
		100-1000	XX0
Total Phosphorus (Automated)	0.01 mg/l	0.01-0.1	0.0X
		0.1-1.0	0.X
		1.0-10	X.X
		10-100	XX
Ortho Phosphorus	0.01 mg/l	0.01-0.10	0.0X
		0.10-1.0	0.XX
		1.0-10	X.X
		10-100	XX
		100-1000	XX0
Silica (ICP)	1 mg/l	1-10	X
		10-100	XX
		100-1000	XX0
		1,000-10,000	XX00
Solids, Total Total, Vol. Suspended Suspended Vol.	1 mg/l	1-10	X
		10-100	XX
		100-1000	XXX
		1,000-10,000	XXX0
		10,000-100,000	XXX00
Solids Settleable	0.1 ml/l	0.1-1.0	0.X
		1.0-10	X.X
		10-100	XX
		100-1000	XX0
		1,000-10,000	XX00
Specific Conductance	0.1 umho/cm	0.1-1.0	0.X
		1.0-10	X.X
		10-100	XX
		100-1000	XXX
		1,000-10,000	XXX0
Sulfate	1 mg/l	1-10	X
		10-100	XX
		100-1000	XXX
		1,000-10,000	XXX0

TABLE 12-1 (cont.)

REPORT OF RESULTS  
TABLE OF SIGNIFICANT FIGURES

Parameter	Minimum Detection Limit	Range	Significant Figures
Sulfide	0.1* mg/l	0.1-1.0 1.0-10 10-100 100-1000	0.X X.X XX XX0
Sulfite	1* mg/l	1-10 10-100 100-1000	X XX XX0
Surfactants	0.05 mg/l	0.05-0.10 0.10-1.0 1.0-10 10-100 100-1000	0.0X 0.XX X.X XX XX0
Turbidity	0.1 NTU	0.1-1.0 1.0-10 10-100 100-1000 1,000-10,000	0.X X.X XX XX0 XX00
Volatile Organics	Varies; see published limits	0.1-1.0 1.0-10 10-100 100-1000	0.X X.X XX XXX

\*Sample volume dependent

### **13.0 SAMPLE COLLECTION, CONTAINERS AND PRESERVATION**

In order to maintain the integrity of the sample from the time it is collected until it is received in the laboratory for analysis, we recommend the use of proper sample containers and preservatives to our clients for the collection of samples. Upon request, we will supply our clients with appropriate sample containers and preservatives. Instructions for sample preservation are given as are material safety data sheets for each preservative supplied.

Certain analyses must be completed in a timely fashion in order to insure that true concentrations are measured. The method of preservation along with the type and volume of preservative used for each parameter is given in Table 13-1. The recommended holding periods are also contained in Table 13-1 for each parameter.

The analyses of those parameters with short holding periods -- 24 hours or less -- are given priority and completed as quickly as possible.

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TABLE 13-1  
SAMPLE PRESERVATION

Parameter	Minimum Recommended Sample Volume ml	U.S. EPA Recommended Preservation Method	Recommended Container Type	U.S. EPA Recommended Holding Time
Acidity-Alkalinity	200	Refrigeration at 4°C	Plastic, Glass	14 Days
Benzene*	2 each, 40	HCl to pH 2 Refrigeration at 4°C	Glass, Teflon Cap	14 Days
Biochemical Oxygen Demand (BOD)	500	Refrigeration at 4°C	Plastic, Glass	48 Hours
Boron	100	Refrigeration at 4°C	Plastic	---
-55- Bromide	100	Refrigeration at 4°C	Plastic, Glass	28 Days
Calcium	200	HNO <sub>3</sub> to pH 2	Plastic, Glass	6 Months
Chemical Oxygen Demand (COD)	200	Refrigeration at 4°C H <sub>2</sub> SO <sub>4</sub> to pH 2	Plastic, Glass	28 Days
Chloride	100	None Required	Plastic, Glass	28 Days
Color	100	Refrigeration at 4°C	Plastic, Glass	48 Hours
Cyanide	500	NaOH to pH 12 and refrigeration at 4°C 2 ml thiosulfate for chlorinated waters	Plastic, Glass	14 Days

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TABLE 13-1 (cont.)

## SAMPLE PRESERVATION

Parameter	Minimum Recommended Sample Volume ml	U.S. EPA Recommended Preservation Method	Recommended Container Type	U.S. EPA Recommended Holding Time
Dissolved Oxygen Winkler	300	Fix on Site	Glass	8 Hours
Ethyl Benzene*	2 each, 40	HCl to pH 2 Refrigeration at 4°C	Glass, Teflon Cap	14 Days
Fluoride	500	None Required	Plastic	28 Days
Hardness	200	HNO <sub>3</sub> to pH to >2	Plastic, Glass	6 Months
Iodide	200	Refrigeration at 4°C	Plastic, Glass	24 Hours
MBAS	250	Refrigeration at 4°C	Plastic, Glass	24 Hours
Metals, Total	100	HNO <sub>3</sub> to pH <2	Plastic, Glass	6 Months
Hexavalent Chromium	500	Refrigeration at 4°C	Plastic, Glass	48 Hours
Metals, Dissolved	100	Filtrate: HNO <sub>3</sub> to pH <2	Plastic, Glass	6 Months
Nitrogen, Ammonia	200	H <sub>2</sub> SO <sub>4</sub> to pH <2	Plastic, Glass	28 Days
Nitrogen, Kjeldahl	100	Refrigeration at 4°C H <sub>2</sub> SO <sub>4</sub> to pH <2	Plastic, Glass	28 Days
Oil and Grease	1 liter	Refrigeration at 4°C H <sub>2</sub> SO <sub>4</sub> to pH <2	Glass	28 Days

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TABLE 13-1 (cont.)

## SAMPLE PRESERVATION

Parameter	Minimum Recommended Sample Volume ml	U.S. EPA Recommended - Preservation Method	Recommended Container Type	U.S. EPA Recommended Holding Time
Organic Carbon	100	Refrigeration at 4°C H <sub>2</sub> SO <sub>4</sub> to pH <2	Plastic, Glass	28 Days
Petroleum Hydrocarbons				
1) EPA Method 418.1	1) 1000	1) HCl to pH 2 and refrigerate	Glass	28 Days
2) California Method	2) 1000	2) Refrigeration at 4°C	Glass	14 Days
3) SW-846 #8015	3) 2 - 40	3) HCl to pH 2, Refrigeration at 4°C	Glass	14 Days
pH	50	Determine on Site	Plastic, Glass	2 Hours
Phenolics	1 liter	Refrigeration at 4°C H <sub>2</sub> SO <sub>4</sub> to pH <2	Glass, Plastic	28 Days
Phosphorus	100	Refrigeration at 4°C H <sub>2</sub> SO <sub>4</sub> to pH <2	Plastic, Glass	28 Days
Ortho-Phosphates	100	Refrigeration at 4°C	Plastic, Glass	48 Hours
Silica	100	Refrigeration at 4°C	Plastic, Glass	28 Days
Specific Conductance	100	Refrigeration at 4°C	Plastic, Glass	28 Days
Solids	500-1000	Refrigeration at 4°C	Plastic, Glass	7 Days
Sulfate	200	Refrigeration at 4°C	Plastic, Glass	28 Days
Sulfide	200	Refrigeration at 4°C 2 ml Zinc Acetate	Glass, Plastic	28 Days
Sulfite	50	Refrigeration at 4°C	Glass, Plastic	48 Hours
Surfactants	500	Refrigeration at 4°C	Plastic, Glass	48 Hours



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TABLE 13-1 (cont.)

## SAMPLE PRESERVATION

Parameter	Minimum Recommended Sample Volume ml	U.S. EPA Recommended - Preservation Method	Recommended Container Type	U.S. EPA Recommended Holding Time
Tannin-Lignin	100	Refrigeration at 4°C	Plastic, Glass	---
Threshold Odor	1 liter	Refrigeration at 4°C	Glass	24 Hours
Toluene*	2 each, 40	HCl to pH 2 Refrigeration at 4°C	Glass, Teflon Cap	14 Days
Turbidity	100	Refrigeration at 4°C	Plastic, Glass	48 Hours
Volatile Organics in Drinking Water*	2 each, 40	HCl to pH 2 Refrigeration at 4°C	Glass	14 Days
Xylenes*	2 each, 40	HCl to pH Refrigeration at 4°C	Glass, Teflon Cap	14 Days

\*For chlorinated waters, the addition of 0.008% sodium thiosulfate is required.

## 14.0 EQUIPMENT LIST

Following is a list of equipment currently used by the Chemical and Industrial Hygiene Division. Notes are included as to make, model, and generalized calibration requirements.

<u>Type</u>	<u>Model</u>	<u>Manufacturer</u>	<u>Calibration Frequency</u>
Oven	C-140G	Blue M	Quarterly
Oven	Muffle Furnace	Hythermoco	Quarterly
Oven	----	S/W	Each Use
Oven	----	Blue M	Each Use
Oven	Moisture Oven	Boeckel	Each Use
Oven	Incubator 0968	Chicago	Each Use
Balance	A-30	Mettler	Daily
Balance	H6T	Mettler	Monthly
Balance	( )	Ohaus	Monthly
Balance	XAD	Fisher	Monthly
Bomb Colorimeter	1241	Parr	Monthly
Spectrophotometer	Spectronic 200	Bausch & Lomb	Each Use
Spectrophotometer	Turbidimeter	HF	Each Use
Centrifuge	----	Drucker	None
Shaker	----	Eberbach	None
Shaker	----	Eberbach	None
Air Compressor	----	Sanborn	None
Emission Spectrophotometer	JY48	Jobin Y-Von	Each Use
Atomic Absorption Spectrophotometer	951	IL	Each Use
Atomic Absorption Spectrophotometer	Video 2200	Thermo Jarrell Ash	Each Use
Microscope	BHT	Olympus	Each Use
Microscope	----	AO	Each Use
Conductivity Bridge	310A	Beckman	Each Use
pH Meter	501	Orion	Each Use
Gas Chromatograph	5890	Hewlett Packard	Each Use
Auto Analyzer	SMAC II	Technicon	Each Use
Auto Analyzer	Boron #1	Technicon	Each Use
Auto Analyzer	Ammonia #2	Technicon	Each Use
Auto Analyzer	Phosphorus #3	Technicon	Each Use
Auto Analyzer	Phosphorus #4	Technicon	Each Use
Oven	Drying Oven	SGA	Each Use
Oven	Drying Oven	Despatch	Each Use

## **15.0 AUDIT PROGRAMS**

Chen-Northern participates in as many performance audit programs as is economically feasible. These audits can take the form of simple round robin analyses or formal audits by regulatory agencies. The following is a list of audit programs in which we are currently enrolled:

- ♦ EPA Safe Drinking Water Analysis Certification
- ♦ EPA NPDES Performance Audit Program for Wastewater Analysis
- ♦ Utah State University Round Robin Soil Analysis
- ♦ NBS Asbestos Identification Round Robin for Bulk Material Analysis

NIOSH PAT Program for Fiber Analysis by Phase Contrast Microscopy

EPA Lead in Airborne Particulate for HiVol Filter Analysis

## **16.0 RECIPIENTS OF QUALITY ASSURANCE PLAN**

1. Montana Department of Health & Environmental Sciences
2. Pat Bugosh - Chen-Northern, Inc. - Helena, MT
3. Ed Hart with Montana Power Company - Butte, MT
4. Lab Staff - Chen-Northern, Inc. - Billings, MT
5. Kathie Roos with Special Resource Management
6. Debbie Madison with Fort Peck Tribes - Poplar, MT
7. Morrison-Maierle/CSSA - Bozeman, MT
8. Joe Nosek - Twin City Testing - St. Paul, MN
9. Chen-Northern, Inc. - Denver, CO
- 10.
- 11.
- 12.
- 13.
- 14.
- 15.

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DANIEL B. STEPHENS & ASSOCIATES, INC.

ENVIRONMENTAL SCIENTISTS AND ENGINEERS

### **Quality Control Program**

Analytical chemistry laboratory services for the IWD RI/FS will be provided by Wadsworth Alert Laboratory. Chen-Northern will perform the laboratory work associated with the geochemical analyses discussed in the hydrogeological investigation. Daniel B. Stephens and Associates will perform the physical property analysis work.

**Insert this section in the back of Appendix D Part B**



## 1. OVERVIEW OF THE PROGRAM

### 1.1 Organizational Objectives

The objectives of the quality assurance/quality control (QA/QC) program of the organization are:

- (1) The generation of data and reports of known quality and integrity.
- (2) Operation of a system to ensure quality in data review, validation, and reporting.
- (3) Operation of a quality assurance management system which provides both quality and task-oriented flexibility.

### 1.2 Responsibilities of Laboratory Personnel

- President of DBS&A  
Designee: Daniel B. Stephens  
Responsibilities:
  - Final approval of Laboratory Report
- Company QA/QC Director  
Responsibilities:
  - Development and implementation of Company QA/QC program
  - Identification of QA/QC needs
  - Direction of QA/QC audits
- Laboratory Manager  
Responsibilities:
  - Implementation of Lab QA/QC program
  - Maintain QA/QC documents
  - Client contact



- Cost estimation of laboratory contracts
  - Deadline
  - Report writing
  - Designing laboratory equipment
  - Explanation of laboratory capabilities
  - Writing computer analysis programs
  - QA/QC final lab reports
- Laboratory Safety Officer  
Responsibilities:
  - Responsible for personnel and laboratory radiation monitoring program
  - Conducting Right-to-Know (Hazard Communication Standard) seminars
  - Updating Material Safety Data Sheets
- Laboratory Assistant Manager  
Responsibilities:
  - Organizing laboratory contract analysis
  - Conducting/supervising laboratory analysis
  - Checking raw data
  - Organization of raw data to computer printout and graphs
  - Organizing draft data report
- Laboratory Analysts  
Responsibilities:
  - Performing/assisting laboratory analysis as directed by the laboratory manager or assistant manager
  - Conducting laboratory equipment maintenance and calibration
  - General cleaning of the laboratory area

### 1.3 Summary of Specific Requirements, QA/QC Program

#### (1) Sample Custody, Identification, Tracking, and Storage Procedures





- a. Chain of Custody
  - b. *Procedures for tracking samples*
  - c. Storage of sample material including materials contaminated with radioactive waste
- (2) Data Collection
- a. Type of data collected
  - b. Methods of data collection
  - c. Accuracy/precision limits of data collected
- (3) Data and Document Handling Procedures
- a. Information system for data and documents
- (4) Data Reduction Procedures
- a. Data input checks
- (5) Analytical, Numerical Analysis
- a. Verification of the basis of the method of analysis
  - b. Check of all hand calculations by a second party
  - c. Independent check of the results of all calculations for their reasonableness, to include, if necessary, an independent partial analysis
  - d. Documentation of all numerical codes
  - e. Verification of all numerical codes (whether internal or public domain) each time they are used, with any change to the code verified by the author and run against either test cases or hand calculations
- (6) Report Review
- a. Check to ensure contractual obligation is fulfilled
  - b. Verification that all QA/QC requirements are met
  - c. Verification of correctness of final report copies



- (7) Performance of Systems and Personnel
  - a. Equipment calibration and maintenance program
  - b. Personnel training and evaluation
  
- (8) Quality Assurance Audits
  - a. Control of nonconformances
  - b. Corrective action
  
- (9) Quality Assurance Records
  - a. Archive system



## 2. SPECIFIC REQUIREMENTS OF THE QA/QC PROGRAM

### 2.1 Sample Custody, Identification, Tracking, and Storage Procedures

All samples which have been collected and transported under a chain-of-custody system will be accepted by DBS&A's laboratory custodian (or designee) following a set of standard operating procedures as described below. The laboratory custodian or designee must carefully check the identification information (e.g., sample number, depth interval, and any other descriptive information) on each sample (either written on the sample container or on a sample tag) with the identification information on the chain-of-custody forms to ensure that all samples are accounted for. If discrepancies exist between a particular sample and the chain-of-custody form, DBS&A's laboratory custodian or designee will note this clearly on the form before signing the custody forms and accepting the shipment of samples. The original chain-of-custody will be kept in the Laboratory Project File created for the samples and their testing program as long as the samples are in the custody of DBS&A. DBS&A will forward copies of the chain-of-custody forms to the client if requested.

After the required testing program is completed, samples will be shipped back to the client together with the chain-of-custody form. The same procedure of checking the information on each sample with that on the custody forms, as well as noting any changes in sample descriptions, will be followed when relinquishing samples.

All sample identification information together with all tests requested by the client for each sample will be legibly recorded on a form entitled "Job Schedule" (see Appendix D for a generic Job Schedule Form). If a DBS&A laboratory number is assigned to samples (for the sake of convenience), the DBS&A number will be written on this form adjacent to the identification information received from the client. All information on this form will be checked, initialed and dated by the form originator as well as one additional member of the laboratory staff.



Two photocopies of the form will be made. One of these copies will be placed in the QA/QC packet of a data binder which will contain all laboratory data sheets as well as the QA/QC information. The other copy will be posted in the laboratory for easy reference. The original will be filed in the Laboratory Project File created for the testing program requested by the client.

The Job Schedule form in the QA/QC packet will be used to track samples through the testing program requested by the client. As each test or task is completed on a sample, it will be noted on the Job Schedule form.

Sample identification numbers will be clearly marked on each sample. If a sample is transferred to another sample container, the sample identification number will be carefully copied to the new sample container.

All original sample material received from a client which is not being subjected to laboratory tests will be stored together in a clearly marked area. All sample material containing ionizing radiation which is not being subjected to laboratory tests shall be stored in a separate storage area away from the main laboratory area. This radioactive sample material will be stored in a restricted storage area if either of the following criteria are met:

- radiation levels which, if an individual were continuously present in the area, could result in his receiving a dose in excess of 2 mrem in any one hour
- radiation levels which, if an individual were continuously present in the area, could result in his receiving a dose in excess of 100 mrem in any seven consecutive days

The laboratory itself will also be considered a restricted area if the above criteria are met during sample testing of radioactive samples. Restricted areas shall be conspicuously posted with a sign or signs bearing the conventional radiation caution colors (magenta or purple on a yellow background).



## 2.2 Laboratory Methods and Data Collection

Laboratory procedures are based on published standards or documentation available in the literature. The procedures used for each test performed in our laboratory are presented as Appendix A, "Laboratory Procedures and Methods".

The type of data collected for each type of laboratory test performed is set forth under "Laboratory Methods" (Appendix A). In general, the method of laboratory analysis used to perform a test will dictate the type of data to be collected. Since explicit written laboratory procedures and methods are based upon documentation available in the literature, such as the ASTM standards or other recognized publications, the type of data collected is dictated by that standard or published method.

Methods of data collection follow standard laboratory practice. Each employee is trained in the basic techniques of reading values from gauges, burets, balances, and other laboratory equipment. In addition, each employee is specifically trained in each method by a senior technical staff member. This training documentation is archived in the laboratory QA/QC file system and is available for inspection by auditors. The senior technical staff trainer certifies that the technician has received the appropriate training. The technician also certifies that he/she has received and understands methods of basic data collection.

Laboratory measurements of mass, temperature, and pressure are dependent on the accuracy of instruments or equipment. Accuracy of our laboratory equipment relies on a rigorous program of calibration, and equipment used to make these measurements is calibrated on a regular schedule. These instruments and typical measurement ranges are listed below.

- Mass measured with electronic balance: 0.001 g to 200 g, 0.01 g to 4000 g
- Temperature measured by mercury in glass thermometer: 1.0° C to 110° C



- *Pressure* measured by gas pressure gauges: 0.005 bars to 3 bars, 0.01 bars to 1.6 bars, 0.01 bars to 4 bars, 0.01 bars to 10 bars, 0.1 bars to 20 bars.

Measurements which depend on limitations in the reading of the instrument are volume and length. The uncertainties associated with measurements of volume and length depend on the piece of equipment being used for measurement. The following uncertainties are typical for the types of equipment listed:

INSTRUMENT	TYPICAL UNCERTAINTY
100-ml graduated cylinder	+ 0.2 ml
10-ml graduated cylinder	+ 0.1 ml
50-ml buret	+ 0.02 ml
Steel ruler (graduated to 1 mm)	+ 1.0 mm

Uncertainties for specific equipment not listed above are:

INSTRUMENT	TYPICAL UNCERTAINTY
Thermometer (10° C to 100° C graduated to 1° C)	+ 0.2° C

### 2.3 Control of QA/QC Documents, Laboratory Data, and Data Reports

All QA/QC documents, laboratory data, and lab data reports are subject to document control procedures outlined in the following. Specific control procedures differ depending on the item being controlled.

QA/QC documents are defined as: this QA Manual, including technical methods of analyses (SOPs); QA/QC forms and records (QA/QC packets) which accompany a data set through laboratory analyses and report preparation; and finally, forms that document any changes to the above-mentioned QA/QC documents. Laboratory data is defined as all data generated during laboratory tests specified by the client. Lab data reports include the raw laboratory data, summary data tables and figures, a discussion of the reasonableness and consistency of the data, and a list of method references used in the client-specified testing program.



The preparation, issue, and change of these QA/QC documents which specify quality requirements or prescribe activities affecting quality must be controlled to assure correct documents are being employed. In addition, these documents must be reviewed for adequacy and approved for release by authorized personnel. The paragraphs below describe how changes are made to documents, who reviews and approves these changes, how often documents are routinely reviewed to ensure quality procedures are up to date and reflect industry standards, and how documents are stored and archived for future reference. In addition, the following describes how data and data reports are handled, stored, and archived.

Changes in QA/QC documents can only be made by or under the direct supervision of the laboratory manager and/or the president of DBS&A. All changes must be reviewed and approved by both the laboratory manager and the president of DBS&A. All changes must be clearly described and approved on a QA/QC Document Revision Form. The old (non-revised) portion of the QA/QC document shall be archived together with the revised portion of the QA/QC document in the laboratory's QA/QC files. The date of this revision shall be clearly marked on these documents, and all revisions shall be filed under document type according to date. These revisions shall be archived for a minimum of five years from their origination.

All original raw laboratory data, together with QA/QC packets (if applicable), for each client-specified testing program are archived in a dedicated data binder in the laboratory office in bookshelves. At least one copy of the final laboratory report, including raw laboratory data in spreadsheet form, summary tables and plots, and written text concerning the consistency and reasonableness of data, are also stored in the laboratory on bookshelves. In addition, a magnetic disk copy of the final laboratory report is stored in the word processing office.

Original data, final data reports, and magnetic disks are all filed according to project number. The client's name also appears on these documents. DBS&A will archive these data and documents for a period of at least five years. After five years, the president of DBS&A and the laboratory manager will decide on final disposition of data and documents; magnetic media will be maintained indefinitely. All non-laboratory personnel must obtain permission from the president of DBS&A or the laboratory manager to view these documents. If the client requests, DBS&A will store the information in a locked file cabinet. The client will be charged extra for secure storage.



A formal system has been developed for handling and controlling laboratory data, QA/QC packets, and laboratory data reports. This system consists of a structured entry, exit, and archive point for all of the above information. This system is described clearly in flow chart form in Appendix B to this QA Manual.

Finally, all data, QA/QC packets, and data reports are considered confidential. Only persons authorized by the laboratory manager or the president of DBS&A are permitted to view, handle, transport, or analyze data obtained through client-specified testing. Unauthorized personnel must have written and verified permission from the client before they will be allowed to have access to testing data.

## **2.4 Data Reduction Procedures**

Data are transferred from the laboratory data sheets, which contain the raw data, to computer files by manual entry through the keyboard. Every datum in the computer file is checked against the raw data sheet by a second party before the report review process is begun.

## **2.5 Analytical, Numerical Analysis**

For a method of analysis not verified in the literature, or not published as a standard method of analysis by a recognized organization (such as ASTM), in-house verification will be performed. Verification of a method of analysis will rely on utilization of accepted principles and concepts of soil physics as they are presently understood. Laboratory procedures, raw data, methods of analysis, and results will be made available to any interested party who wishes to examine an in-house verification.

Results of any analysis that relies on hand calculations is checked by a second party.

All calculations, whether performed by computer or by hand, are examined by a second party for their reasonableness.





Documentation of numerical codes is maintained on open file and is available for inspection by any interested party with approval by the president and laboratory manager of DBS&A. For numerical codes that have been written in-house, documentation may consist of published articles in recognized journals, in-house analytical analysis, or both. For all codes, test cases with known published analytical solutions are utilized as a primary tool in the verification procedure.

Prior to each use, numerical codes are checked against a test case.

## **2.6 Report Review**

Final reports are thoroughly checked by both the laboratory manager and the president of DBS&A. In addition to verifying the correctness of every reported value, the report is checked for completion of contractual obligation and completion of all QA/QC requirements. Verification of completion of QA/QC requirements is done by examination of the QA/QC reporting forms that accompany the raw data and final report.

## **2.7 Performance of Systems and Personnel**

Instrument and equipment performance is assured by a documented program of calibration and maintenance. Performance of personnel is assured by hiring only qualified personnel who have high academic achievement records and who have previous laboratory experience (minimum of college chemistry or equivalent), and by training laboratory personnel in each laboratory method to be performed. Resumes which document academic and other training and experience are kept on file for all personnel. Records are also kept on file which document training of personnel in specific laboratory methods used at DBS&A. In addition, laboratory personnel are encouraged to attend relevant conferences and training seminars, and the cost of such outside training is frequently borne by DBS&A.

## **2.8 Quality Assurance Audits, Nonconformances, and Corrective Action**

Random QA/QC system and personnel audits or checks are made periodically by the QA/QC Program Director. A minimum of one internal audit will be performed annually. A system and/o

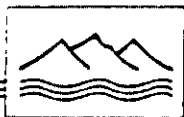


personnel QA/QC audit may be performed at any time by the client's QA/QC personnel with the authorization of DBS&A's president, QA/QC director, or laboratory manager. Prior to any audit, QA/QC Form 015b, Audit Plan, will be completed by the lead auditor. An audit checklist (QA/QC Form 015C) will be utilized for all audits performed. These records will be maintained by the QA/QC director and the laboratory manager.

All QA/QC audit team personnel will be certified prior to participating in audits. This certification will be registered on QA Form 014. The record of certification will be maintained by the QA/QC director and the laboratory manager.

If an audit determines that the laboratory is not conforming to any component of the QA/QC program as defined in this document (Quality Assurance and Quality Control Program), a written Quality Finding Report statement (QA Form 015A) describing the nonconformance(s) shall be generated by the auditor and delivered to the laboratory manager and the QA/QC program director (if applicable). The laboratory manager will have one week to take corrective action to eliminate the nonconformance and to document in writing the nature of the corrective action. All Quality Finding Reports will be logged in the Quality Finding Report Log.

Laboratory measurement equipment or analytical methods that fail to meet project QC requirements will be immediately brought to the attention of the laboratory manager. If failure is due to equipment malfunction, the equipment will be repaired and recalibrated, and the analysis will be repeated. Every attempt will be made to repeat all affected parts of the analysis so that data will not be affected by failure to meet QC requirements. Nonconforming data will be qualified with a note specifying reasons for the qualification. All incidents of failure to meet QC requirements and all corrective actions taken will be documented and placed in appropriate project files. Deficiencies noted during checks of raw data will be immediately corrected. This action will vary depending upon problems noted and can range from correcting miscalculated data to requiring the re-analysis of samples. Documentation of corrective action measures will be made available to the QA/QC director. Corrective action documentation will include the following information (where applicable):



- Nature of the problem
- Date and time of discovery
- Analytical parameter(s) affected
- Sample lot affected
- Date, time, and description of the resulting corrective action
- Signature of the laboratory QC manager

The laboratory manager will prepare a written summary on corrective actions for the client. This summary will review the validity, quality, and completeness of the data in question and, as necessary, make recommendations for corrective action, e.g., further sampling or additional analyses. Corrective action will be implemented when the project objectives are not met or when conditions adverse to quality have been identified. Conditions adverse to quality shall be promptly identified and corrected as soon as possible. The identification, cause, and corrective actions to prevent recurrence shall be determined and documented for significant conditions adverse to quality. All corrective action requests will be logged in the corrective action request log.

## **2.9 Quality Assurance Records**

QA/QC records pertaining to the collection, analysis, and reporting of data remain as a permanent part of the original data set in open file.

QA/QC records pertaining to the verification of laboratory procedures and numerical codes are kept on open file. QA/QC records pertaining to the calibration of systems and equipment are maintained as a part of the calibration open file.

QA/QC records covering the training of personnel are also kept in an open file. Records concerning the performance of laboratory personnel are confidential and require the authorization of the president of DBS&A to be examined by persons other than the laboratory manager.

QA/QC records documenting and approving changes in any QA/QC document shall be stored in the QA/QC file.



### 3. IMPLEMENTATION OF THE QA/QC PROGRAM

The principle areas covered by QA/QC procedures are 1) sample custody, identification, and tracking; 2) instrumentation and equipment performance; 3) laboratory testing methods and the recording, reduction, and analysis of the data obtained from such testing; 4) personnel training; 5) QA/QC audits, nonconformances, and corrective action; and 6) document control. Specific procedures and forms have been developed by DBS&A to ensure that quality is maintained in all these areas. The specific reporting mechanisms which form the basis of the QA/QC program are described in this section.

#### 3.1 Sample Custody, Identification, and Tracking

The chain-of-custody form which accompanies samples (when utilized by the client) ensures that samples collected in the field are the same samples that will be tested in the laboratory. This form also helps to track down lost samples and to document any sample damage which may have occurred during shipping.

Samples are tracked through a testing program requested by the client by utilizing the Job Schedule form. As each task is completed on each sample, it is noted on the Job Schedule form.

#### 3.2 Instrumentation and Equipment Performance

A written record of equipment and instrument calibration is the primary QA/QC means to ensure performance. This calibration record includes:

- (1) A summary table (see Appendix D) listing all equipment requiring calibration, the interval of time between calibrations, dates of previous calibrations, and dates of the next required calibrations.
- (2) Calibration data sheets from each (NBS traceable) calibration conducted on each piece of equipment.



As a minimum, calibration data to be provided from the analytical laboratory shall include:

- Type of equipment used and the detection limits for the equipment.
- Calibration method and sequential actions.
- Calibration data regarding form and format.
- List of primary and secondary standards used.
- Continuing calibration control charts.
- A list of critical or replacement parts.

Each piece of equipment shall be identified such that the pertinent calibration information can be retrieved. The equipment shall have an individual calibration log and be calibrated/standardized prior to use or as a part of the operational use following the manufacturer's recommended procedures for calibration/standardization.

Measuring and test equipment shall be calibrated at prescribed intervals and/or prior to use. Frequency shall be based upon the type of equipment, inherent stability, manufacturer's recommendations, intended use and experience.

Responsibility for calibration rests with the laboratory manager. It is the responsibility of the personnel using the equipment to check the calibration status prior to using it to ensure the equipment is operational.

Records shall be prepared and maintained for each piece of calibrated equipment to indicate that established calibration procedures have been followed. Calibration records for the equipment controlled by the various laboratories, offices, and groups shall be maintained by the respective organizations. A copy of the instrument logbook shall be provided to indicate calibration status when the samples were being analyzed.

Calibration Failure: Field and laboratory equipment found to be out of calibration shall be recalibrated in accordance with the requirements of this section. When test equipment is found to be out of calibration, damaged, lost, or stolen, an evaluation shall be made to ascertain the validity of previous inspection or test results and the acceptability of components inspected and/o.



tested since the last calibration check. When it is necessary to assure the acceptability of suspect items, the originally required inspections and/or tests shall be repeated using properly calibrated equipment. Suspect items on which a questionable device was used shall be listed in a nonconformance report or a deficiency notice, as applicable.

Test equipment consistently found to be out of calibration shall be repaired or replaced.

Inspection and test reports shall include identification of the test equipment used to perform the inspection and/or test.

DBS&A also follows a program of regular equipment maintenance to help ensure that equipment performs as intended. Equipment maintenance instructions and schedules are summarized in Appendix D. Most maintenance involves simply "good housekeeping" procedures such as cleaning after each use or as needed. An instrument maintenance log book shall be kept which includes maintenance schedules, instructions, and maintenance history. The laboratory manager shall be responsible for all laboratory preventative maintenance.

Finally, DBS&A relies on their highly competent and trained personnel to closely follow documented methods and recognize when equipment is malfunctioning. A combination of regular equipment calibration, regular maintenance, and operation by trained and competent personnel who follow standard procedures, ensures proper equipment performance and a data product which will stand up to highest level of QA/QC technical review.

### **3.3 Laboratory Testing Methods and the Recording, Reduction, and Analysis of the Data Obtained From Such Testing**

A total of six written records control the QA/QC of laboratory testing methodology and the data obtained during testing. These written records accompany the laboratory data set and are comprised of the following:



- (1) Equipment Calibration Records Summary. This lists all DBS&A equipment requiring calibration, the time interval between calibrations, dates of previous calibrations, and dates of the next required calibrations.
- (2) Check of Computer Programs Used in the Analysis of Laboratory Data. Validation runs are done prior to each use of a computer program. The results of these validation runs accompany the data set.
- (3) Validation of Hand Calculations by a Second Party. All manual calculations are checked by a second party reviewer. The results of this check accompany the data set.
- (4) Data and Report Review Checks. This record is a written and attested verification of the correctness and completeness of the data report, and covers every aspect of the report's content. This verification accompanies the data set.
- (5) QA/QC Recommendations Summary. This form provides a formal means to record problems encountered during laboratory testing and data analysis as well as a means for making recommendations to eliminate these problems. The laboratory manager will discuss all noted problems and recommendations with laboratory personnel and if possible propose a course of action to be taken to eliminate problems in future testing. Proposed actions will be discussed with the QA/QC director prior to implementation. In summary, this form provides an internal mechanism by which the overall laboratory testing and data analysis program can be improved.
- (6) QA/QC Check. This record verifies that all QA/QC requirements for a job have been completed.

One written record that does not accompany the data set, but that falls into this category, is the Documentation and Verification of a Computer Code. The initial documentation and verification of a computer code is performed as a separate task. The computer code, relevant written documentation of the code, and the results of computer runs of test cases are kept in permanent open files.



### 3.4 Personnel Performance

Daniel B. Stephens & Associates, Inc. recruits and hires the most qualified personnel available. These individuals have high academic achievement as well as directly relevant prior experience.

All new laboratory personnel are trained in each laboratory testing method by senior and experienced laboratory staff (e.g., lab manager, assistant lab manager, or an experienced laboratory analyst). This training is documented in a Record of Training form (QA Form 013), which is kept in the laboratory QA files.

Periodic group training sessions are conducted to increase the skill levels of our employees. Attendance at outside training sessions is encouraged, and the cost of such training is often borne by the firm.

To ensure that a high level of competence is maintained, periodic performance reviews are completed. These reviews are done on no less than an annual basis. Input to the review process consists of both written QA/QC records of performance and visual observations of supervisors. Performance reviews are confidential.

### 3.5 QA/QC Audits, Nonconformances, and Corrective Action

All QA/QC audits, nonconformances, and corrective actions must be documented in writing and archived in DBS&A's QA/QC File. At a minimum the QA/QC audit documentation must contain the following: the names, signatures, and affiliations of the auditor(s), the date, and the components of the QA/QC program audited, with any nonconformance(s) clearly detailed. Corrective action documentation initiated by the laboratory manager must at a minimum contain the date, name of the laboratory manager and his signature, the nonconformance(s) as specified in the audit, and a detailed description of corrective action(s) taken and the date these actions were implemented.





### 3.6 Document Control

General procedures for control of QA documents, laboratory data, and laboratory data reports have been outlined in Section 2.3. Provisions for implementing these procedures are described in the following.

Data and report documents are controlled by means of a series of seven forms (QA Forms 1 through 18) contained in the QA/QC packet. These forms are filled out as testing and report preparation is in progress. These forms are archived together with lab data and reports in DBS&A's laboratory office for a minimum of five years.

### 3.7 Laboratory Environmental Parameters

In addition to the foregoing written QA/QC records, the laboratory temperature and relative humidity are recorded on paper strip charts. A copy of the strip charts is kept in an open file.

### 3.8 QA/QC Forms

All forms used in the QA/QC program are presented in Appendix D. These forms are utilized to record, transmit, and archive the required QA/QC information, as outlined in the foregoing sections.